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(54) **METHODE DE PRODUCTION DE DETERGENTS SOUS FORME
SOLIDE**
(54) **METHOD FOR PRODUCING DETERGENT FORMS**

(57) The present invention relates to a process for the production of formed bodies having active washing and cleaning properties. In particular, the invention relates to a process for the production of detergent and cleaning agent formed bodies for washing of textiles in a domestic washing machine, which are, in brief, called detergent tablets. The invention also relates to a process for the production of detergent and cleaning agent formed bodies comprising the steps of a) preparing mainly solid, finely particulate constituents in a plasticised state to form pourable and flowable high density granules, b) preparing one or several agglomerates from further constituents to be optionally added, c) combining the granules and agglomerates of steps a) and b) to form compressed pre-mixtures, and d) compressing the pre-mixtures to form single or multiple phase formed bodies, wherein the granules and agglomerates produced in steps a) and b) are essentially free from fines and have particles sizes of between 800 and 2000 µm.



Abstract

The present invention relates to a process for the production of formed bodies having active washing and cleaning properties. In particular, the invention relates to a process for the production of detergent and cleaning agent formed bodies for washing of textiles in a domestic washing machine, which are, in brief, called detergent tablets.

5 The invention also relates to a process for the production of detergent and cleaning agent formed bodies comprising the steps of a) preparing mainly solid, finely particulate constituents in a plasticised state to form pourable and flowable high density granules, b) preparing one or several agglomerates from further constituents to be optionally added, c) combining the granules and agglomerates of steps a) and b)

10 to form compressed pre-mixtures, and d) compressing the pre-mixtures to form single or multiple phase formed bodies, wherein the granules and agglomerates produced in steps a) and b) are essentially free from fines and have particles sizes of between 800 and 2000 μm .

METHOD FOR PRODUCING DETERGENT FORMS

Field of the Invention

5 The present invention relates to a process for the production of formed bodies having active washing and cleaning properties. In particular, the invention relates to a process for the production of detergent and cleaning agent formed bodies for washing of textiles in a domestic washing machine, which are, in brief, called detergent tablets.

Background of the Invention

10 Nowadays, commercial detergents and cleaning agents are available in the form of liquid products or solids. As regards the latter type, a distinction is made between conventional powders and concentrates which may be obtained by granulation or extrusion, for example. Compared with the conventional powders, concentrated detergents and cleaning agents have the advantage of a reduced amount of packaging
15 having to be dealt with and quantitatively less product has to be metered per wash cycle. Moreover, as a result of the reduced packet sizes, transport and storage costs are reduced. The most highly concentrated form in which detergents and cleaning agents are at present on offer on the market in some countries consist of compressed detergent and cleaning agent formed bodies. Whereas water softeners and
20 dishwashing agents for dishwashers are widely available in this form, numerous problems have occurred in detergents for textiles, which have so far prevented wide distribution and acceptance by the consumers. As a result of the substantially higher surfactant contents, the problems, which usually occur in the case of the formed body form available, are further increased. Particular problems are posed by detergents
25 tablets containing alkoxylated non-ionic surfactants since these types of surfactant have a negative effect on the solubility of the tablets – whereas, on the other hand, it is these surfactants which are specifically desired because of their high level of detergency.

30 The dichotomy between a sufficiently hard formed body and a sufficiently rapid decomposition time, in particular, presents a central problem in this respect. Since sufficiently stable, i.e. dimensionally stable formed bodies resistant to fracturing can

be produced only by way of relatively high compression pressures, a strong compaction of the formed body components and consequently a delayed disintegration of the formed body in the aqueous liquor and thus an excessively slow liberation of the active substances in the washing and cleaning process occurs. In addition, the delayed disintegration of the formed body has a disadvantage that the usual detergent and cleaning agent formed bodies cannot be flushed into domestic washing machines through the dispenser chamber since the tablets do not break down sufficiently rapidly into secondary particles small enough to be flushed from the dispenser chamber into the washing machine drum.

To solve this problem, various approaches have been made in the state of the art. Apart from using special constituents intended to promote disintegration, coating individual constituents and sieving of the pre-mixtures to be compressed have been suggested.

Thus, **EP-A-0 466 484** (Unilever) discloses detergent tablets in the case of which the pre-mixture to be compressed has particle sizes of between 200 and 1200 μm , the upper and lower limits of the particle sizes differing by no more than 700 μm . Compressing substantially coarser particles into tablets is not suggested in this specification.

EP-A-0 522 766 (Unilever) also relates to formed bodies of a compacted, particulate detergent composition containing surfactants, builders and disintegration aids (e.g. those based on cellulose), at least part of the particles being coated with the disintegration aid which exhibits both a binding and a disintegration effect during dissolution of the formed bodies in water. This specification also points out the general problems involved in producing formed bodies of adequate stability and a simultaneous satisfactory solubility. The particle size in the mixture to be compressed should in this case be above 200 μm , the upper and lower limit of the individual particle sizes not differing by more than 700 μm . In this specification, to, it is explicitly stated that the particles should not be coarser than 1200 μm .

DE 40 10 533 (Henkel KGaA) discloses a process for the production of compressed bodies made from pre-compacted granules. In this case, the granules previously produced in a first operating stage by extrusion moulding and comminution are, if necessary, mixed with further constituents and auxiliary agents and tableted. The proportion of pre-compacted granules in the compressed bodies is as much as 100% in this specification. A pretreatment of the optionally used components to be admixed is not disclosed.

10 — Although the detergent tables produced according to the above documents have a sufficient hardness, they exhibit decomposition rates which do not allow metering via a dispenser chamber in a domestic washing machine. In the above-mentioned specifications of the state of the art, dissolution times of less than 10 minutes and residual values of less than 50% are considered satisfactory, such values being
15 completely unsatisfactory for the use of detergent tablets via the dispenser chamber.

A further disadvantage of the formed bodies produced according to the state of the art consists of their unsatisfactory resistance to shock type impact. The tablets are sufficiently stable vis-à-vis pressure which is exerted on them on a slowly increasing
20 scale but burst when being dropped onto a hard substrate, for example. When being dropped or transported, it is also possible for edge breakage phenomena to occur on the tablets against which conventional tablets are insufficiently resistant. In addition, the usual detergent and cleaning agent formed bodies have the disadvantage of hardening further or becoming deliquescent during storage, so that they must be
25 protected against the surrounding air – something that is usually achieved by individual packaging.

Summary of the Invention

Consequently, the present invention was based on the task of providing a process for
30 the production of detergent and cleaning agent formed bodies, which process makes it possible to produce formed bodies which are free from the above-mentioned disadvantages. In this process, it should be possible to produce detergent and

cleaning agent formed bodies in a simple and highly reproducible manner, which formed bodies have a high level of hardness, are characterised by a rapid rate of dissolution and can be used via the dispenser chamber in domestic washing machines. In this respect, the hardness should be restricted not only to a high diametral fracture stress, the stability of the formed bodies should be guaranteed during transportation (friction/vibration stress) and while being dropped.

In addition, the formed bodies to be produced according to the process to be provided should not undergo any changes to their advantageous property profile during open storage so that air-tight packaging of the individual tablets becomes necessary.

It has now been found that detergent and cleaning agent formed bodies with the above-mentioned advantages can be produced by compressing pre-mixtures which contain both pourable and flowable high density granules produced via the plasticised state as well as agglomerates of further optional constituents which have particle sizes of between 800 and 2000 μm and are essentially free from fines.

The subject matter of the invention is a process for the production of detergent and cleaning agent formed bodies, which process comprises the steps

- a) Processing of at least mainly solid, finely particulate constituents in the plasticised state to form pourable and flowable high density granules.
- b) The production of one or several agglomerates from further constituents to be optionally used.
- c) Combining the granules of steps a) and b) to form compressed pre-mixtures and
- d) Compressing the pre-mixtures to form single or multiple phase formed bodies the granules produced in steps a) and b) being essentially free from fines and having particle sizes of between 800 and 2000 μm .

Within the framework of the present invention, the term "essentially free from fines" characterises particle mixtures which exhibit less than 20% by weight of particles of a size of less than 800 μm . In particular, particle mixtures are preferred whose content

of particles of a size of less than 600 μm is below 10% by weight, mixtures being preferred which contain a maximum of 3% by weight of particles with a size of less than 400 μm . Overall, it is preferred to keep the proportion of particles with particle sizes of less than 800 μm even lower, for example below 15% by weight, preferably below 10% by weight and in particular below 5% by weight.

The particles of the pre-mixture which, in step d) of the process according to the invention, are compressed to form detergent and cleaning agent formed bodies have particle sizes of between 800 and 2000 μm . In this respect, it is preferred for at least 75% by weight of these particles to have a particle size of between 800 and 1600 μm . Mixtures of the granules produced in steps a) and b), at least 60% by weight which consists of particles with a particle size of between 1200 and 15 μm , are again preferred.

Detailed Description of the Invention

The process according to the invention is broken down into four steps: in the first step, at least predominantly solid, finely particulate constituents are processed in the plasticised state to form pourable and flowable high density granules. The second step comprises the production of one or more agglomerates from further constituents to be optionally used, the production process being here – as in the first step – adjusted such as to achieve the desired particle size range. The granules/agglomerates produced in the first two steps a) and b) are mixed together in the third step and subsequently compressed to form detergent and cleaning agent formed bodies.

Process step a):

The production of the high density pourable and flowable granules in step a) is effected by processing in the plasticised state. The production processes for such granules are described in the state of the art and can be used within the framework of partial step a) of the process according to the invention.

A process to be preferably used for the production of pourable and flowable high density granules via the plasticised state is described in the earlier German patent application 196 38 599.7 (Henkel KGaA). According to the theorem of this application, the production of the pourable and flowable high density granules in step a) is effected by joining detergent or cleaning agent compounds and/or detergent or cleaning agent raw materials with simultaneous or subsequent forming, a solid pre-mixture being initially produced which contains individual raw materials and/or compounds which are present as a solid at room temperature and a pressure of 1 bar and exhibit a melting point or softening point of not less than 45°C and also contains, if-necessary, up to 10% by weight of non-ionic surfactants which are liquid at temperatures below 45°C and a pressure of 1 bar and which, by applying compaction forces at temperatures of at least 45°C, and are converted into a grain and, if necessary, subsequently processed or treated further with the proviso that

- the pre-mixture is essentially anhydrous and
- at least one raw material or compound, which is solid at a pressure of 1 bar and temperatures below 45°C, is present under the processing conditions as a melt, this melt serving as a polyfunctional, water- soluble binder which, during the production of the agents, operates as a slip and as an adhesive for the solid detergent or cleaning agent compounds or raw materials but has a disintegrating effect during the re-dissolution of the agent in the aqueous liquor.

Within the framework of the disclosure of this specification, the term "essentially anhydrous" should be understood to refer to a state in which the content of liquid water, i.e. water not present in the form of water of hydration and/or constitutional water, is below 5% by weight, preferably below 3% by weight and in particular even below 0.5% by weight, based on the pre-mixture. Consequently, water can be introduced into the process for the production of the pre-mixture essentially only in the chemically and/or physically combined form or as a component of the raw materials or compounds present as solids at temperatures below 45°C and at pressure of 1 bar but not as a liquid, solution or dispersion.

In the above-mentioned specification, the term particulate detergents or cleaning agents is understood to mean those agents which do not contain dust-type fractions and, in particular, do not have particle sizes of less than 200 μm . In a particularly preferred embodiment of the invention disclosed therein, the detergents or cleaning agents produced, the compounds or the treated raw materials consist - in a proportion of at least 70% by weight, preferably at least 80% by weight and particularly preferably a higher proportion of up to 100% by weight - of spherical (bead type) particles with a particle size distribution in the case of which at least 80% by weight of the particles are between 0.8 and 2.0 mm.

The term detergents or cleaning agents should be understood to mean those compositions which can be used for washing and cleaning without commonly other constituents having to be admixed. A compound, on the other hand, consists of at least 2 components usually used in detergents or cleaning agents; however, compounds are normally used only in mixture with other components, preferably together with other compounds. Within the framework of this invention, a treated raw material is a relatively finely particulate raw material which is converted by the process according to the invention into coarser particles. Strictly speaking, a treated raw material is a compound within the framework of the invention if the treatment agent is a constituent usually used in detergents or cleaning agents.

The constituents used in the above-mentioned process - with the exception of the non-ionic surfactants liquid at temperatures below 45°C and a pressure of 1 bar, which may be present if necessary - may consist of compounds produced separately but also of raw materials which are present in powder or particulate form (finely divided to coarse) and are, in any case, solid at room temperature and a pressure of 1 bar. Suitable particulate particles which can be used are, for example, beads or (fluid bed) granules etc produced by spray drying. The composition of the compounds as such is unimportant for the invention with the exception of the water content which must be such that the pre-mixture, as defined above, is essentially free from water and contains preferably not more than 10% by weight of water of hydration and/or constitutional water. It is also possible to use solid compounds in the pre-mixture

which compounds serve as carriers for liquids, for example for liquid non-ionic surfactants or silicone oil and/or paraffins. These compounds may contain water to the extent as indicated above, the compounds being flowable and remaining flowable or at least conveyable even at higher temperatures of at least 45°C. In particular, however, it is preferable that compounds with maximum 10% by weight and particularly preferentially with maximum 7% by weight of water, based on the pre-mixture, are used in the pre-mixture. Free water, i.e. water which is not combined in any form with a solid and consequently is present "in the liquid form" is preferably not contained in the pre-mixture since even very small quantities, e.g. approximately 0.2 or 0.5% by weight, based on the pre-mixture, are already sufficient to partially dissolve the binder which is water-soluble as such. This would have the consequence of reducing the melting point or the softening point and causing the end product to lose both some of its flowable properties and some of its bulk density.

It has been found that it is in no way irrelevant to which solid raw material or in which solid compound the water is bound. Thus, water which is bound to builder substances such as zeolite and silicates (details of the description of the substances are given below), in particular if the water is bound to zeolite A, zeolite P or MAP and/or zeolite X, can be considered as being less critical. On the other hand, it is preferred that water which is bound to solid components other than the above-mentioned builder substances should preferably be present in the pre-mixture in quantities of less than 3% by weight. In one embodiment of the invention, it is therefore preferred for the content of bound water in the pre-mixture to be no more than 10% by weight and/or the content of water not bound to zeolite and/or silicates to be less than 7% by weight and in particular maximum 2-5% by weight. In this connection, it is particularly advantageous if the pre-mixture contains no water that is not bound to the builder substances. However, this is difficult to achieve technically since, as a rule, at least traces of water are always entrained by the raw materials and compounds.

The content of liquids which are non aqueous at temperatures below 45°C present in the solid compounds used in the pre-mixture amounts preferably also or additionally to up to 10% by weight, preferably to as much as 6% by weight, again based on the

pre-mixture. In particular, solid compounds are used in the pre-mixture, which compounds contained usual non-ionic surfactants liquid at temperatures below 45°C and a pressure of 1 bar, which compounds can be produced separately according to any of the known types of production – e.g. by spray draying, granulation or atomisation treatment of carrier beads. In this way, pre-mixtures can be produced which allow, for example, up to approximately 10% by weight, preferably less, in particular up to maximum 8% by weight and, for example, between 1 and 5% by weight of non-ionic surfactants, based on the finished agent.

Compounds which contain water in the form as indicated above and/or serve as carrier for liquids, in particular for non-ionic surfactants which are liquid at room temperature, i.e. contain these constituents which are liquid at room temperature and can be used according to the theorem of the above-mentioned application, have under no circumstance a softening point below 45°C. Similarly, the individual raw materials used separately have a melting point of at least 45°C. Preferably, the melting point and/or the softening point of all the individual raw materials and compounds used in the pre-mixture is above 45°C and advantageously at least 50°C.

In a preferred embodiment of the invention, at least 80% by weight, in particular at least 85% by weight and particularly preferably at least 90% by weight of the compounds and individual raw materials used in the pre-mixture have a much higher softening point and/or melting point than those that can be reached under the process conditions. In practise, the process temperatures will not be above 150°C, preferably not above 120°C, for economic reasons alone. Thus, at least 80% by weight of the compounds and individual raw materials used will have a softening point and/or melting point above 150°C. As a rule, the softening point or the melting point is substantially above this temperature. If constituents are used which decompose under the effect of the temperature, e.g. peroxy bleaching agents such as perborate or percarbonate, the decomposition temperature of these constituents at a pressure of 1 bar and in particular at higher pressures which are present in the preferred extrusion processes according to the invention, is also significantly above 45°C.

Apart from the solid components, the pre-mixture can additionally contain up to 10% by weight of non-ionic surfactants liquid at temperatures below 45°C and a pressure of 1 bar, in particular the alkoxylate alcohols usually used in detergents or cleaning agents such as fatty alcohols or oxy alcohols with a C chain length of between 8 and 20 and in particular on average 3 to 7 ethylene oxide units per mole alcohol (a detailed description is given below). The addition of the liquid non-ionic surfactants can be effected in quantities which are such as to ensure that the pre-mixture is still present in the flowable form. If such liquid non-ionic surfactants are introduced into the pre-mixture it is preferable that liquid non-ionic surfactants and the binder having a disintegrating effect are introduced separately into the process. In such a preferred embodiment of the invention, the liquid non-ionic surfactants are applied by means of nozzles in a continuous production process onto the powder stream and absorbed by the latter.

However, the pre-mixture also contains at least one raw material or at least one compound which serves as a binder and which is solid at room temperature but present as a liquid or in the form of a melt during compaction under the conditions of the process. The binder itself can be atomised onto the pre-mixture once it has melted or added dropwise to the pre-mixture; on the other hand, however, it has proved advantageous to introduce the binder in the solid form as a powder into the pre-mixture. The melting point or the softening point at a pressure of 1 bar is at least 45°C and (in particular for economic reasons) preferably below 200°C, in particular below 150°C. If the binder is introduced into the pre-mixture in the form of a melt, the temperature in the melting vessel is also more than 45°C to maximum approximately 200°C, the temperature in the melting vessel being able to significantly exceed the melting temperature or the temperature of the softening point of the binder or the binder mixture.

The type of suitable binder and the temperature in the process step of compaction are dependent upon each other. As it has proved advantageous for the binder in the process step of compaction to be as homogeneously distributed in the compacted substance as possible, temperatures must be present during the process step of

compaction at which the binder softens at least and is preferably present in the completely, and not merely the partially, molten form. If, consequently, a binder with a high melting point or a high softening point is chosen, a temperature must be adjusted during the process step of compaction at which melting of the binder is guaranteed. In addition, it should be possible to process also temperature-sensitive raw materials, depending on the desired composition of the end product. The upper temperature limit in this respect is set by the decomposition temperature of the sensitive raw material, it being preferable to operate significantly below the decomposition temperature of this raw material. On the other hand, the lower limit with respect to the melting point or the softening point is of great importance because, as a rule, an end product is obtained at melting points or softening points below 45°C which tends to agglutinate at temperatures as low as room temperature and at slightly elevated temperatures around 30°C, i.e. at summer temperature and under storage and transport conditions. It has proved to be particularly advantageous to work at a temperature a few degrees, e.g. 2 to 20°C, above the melting point or above the softening point.

Preferred binders which can be used as such or in mixture with other binders are polyethylene glycols, 1,2-polypropylene glycols and modified polyethylene glycols and polypropylene glycols. The modified polyalkylene glycols include in particular the sulphates and/or disulphates of polyethylene glycols or polypropylene glycols with a molecular weight between 600 and 12000 and, in particular, between 1000 and 4000. A further group consists of mono and/or disuccinates of the polyalkylene glycols which, again, have molecular weights of between 600 and 6000, preferably between 1000 and 4000. For a more detailed description of the modified polyalkylene glycol ethers, reference should be made to the disclosure of the International Patent Application WO-A-93/02176. Within the framework of this invention, polyethylene glycols include those polymers for the production of which not only ethylene glycol but also C₃-C₅ glycols as well as glycerine and mixtures of these are used as starting molecules. They also comprise ethoxylated derivatives such as trimethylol propane with 5 to 30 EO.

The polyethylene glycols preferably used may have a linear or branched structure, linear polyethylene glycols being preferred in particular.

5 The polyethylene glycols which are preferred, in particular, include those with molecular weights between 2000 and 12000, advantageously around 4000, it being possible to use polyethylene glycols with molecular weights of less 3500 and above 5000, in particular in combination with polyethylene glycols with a molecular weight of around 4000, such combinations advantageously containing, in a proportion of more than 50% by weight based on the total quantity of the polyethylene glycols,
10 polyethylene glycols with a molecular weight of between 3500 and 5000. However, it is also possible to use polyethylene glycols as binders which, as such, are present in the liquid state at room temperature and a pressure of 1 bar; this involves above all polyethylene glycol with a molecular weight of 200, 400 and 600. However, these polyethylene glycols which are liquid as such, should be used only in mixture with at
15 least one further binder, this mixture having to satisfy again the requirements of the invention, i.e. having a melting point and/or softening point of at least more than 45°C.

20 Low molecular weight polyvinyl pyrrolidones and their derivatives with molecular weights of maximum 30000 are also suitable as binders. In this respect, molecular weight ranges of between 3000 and 30000, preferably around 10000 are preferred. Preferably, polyvinyl pyrrolidones are used not as sole binder but in combination with others, in particular in combination with polyethylene glycols.

25 Suitable further binders have proved to consist of raw materials with active detergent or cleaning agent properties, i.e., for example, non-ionic surfactants with melting points of at least 45°C or mixtures of non-ionic surfactants and other binders. The preferred non-ionic surfactants include alkoxyated fatty or oxoalcohols, in particular C₁₂-C₁₈-alcohols. In this respect, alkoxylation levels, in particular ethoxylation levels
30 of, on average, 18 to 80 AO, in particular EO per mole alcohol, and mixtures thereof have proved to be particularly advantageous. In particular, fatty alcohols with an average of 18 to 35 EO, in particular with an average of 20 to 25 EO, exhibit

advantageous binder properties according to the meaning of the present invention. If necessary, the binder mixtures can also contain ethoxylated alcohols with, on average, fewer EO units per mole alcohol, such as tallow fatty alcohol with 14 EO. However, it is preferable to use these alcohols with a relatively low ethoxylation level only in mixture with alcohols with a higher ethoxylation level. Preferably, the content of binder in these alcohols with a relatively low ethoxylation level is less than 50% by weight, in particular less than 40% by weight, based on the total quantity of binder used. In particular, non-ionic surfactants commonly used in detergents or cleaning agents, such as C₁₂-C₁₈-alcohols with a average of 3 to 7 EO, which are liquid at room temperature, are preferably present in the binder mixtures merely in quantities such that less than 2% by weight of these non-ionic surfactants, based on the end product of the process, are thus provided. As described above, it is, however, less preferable to use non-ionic surfactants liquid at room temperature in the binder mixtures. In a particularly advantageous embodiment, such non-ionic surfactants do not form part of the binder mixture since they reduce not only the softening point of the mixture but may also contribute to the tackiness of the end product and, moreover, as a result of their tendency to undergo gel formation on contact with water do not satisfy the requirements regarding rapid dissolution of the binder/the separation wall in the end product to the desired extent. Similarly, it is not preferable for anion surfactants or their precursors, namely the anionic surfactant acids, which are commonly used in detergents or cleaning agents, to be present in the binder mixture.

Other non-ionic surfactants which are suitable as binders consist of the fatty acid methyl ester ethoxylates which have no gelling tendency, in particular those with an average of 10 to 25 EO (a more accurate description of this group of substances is given below). Particularly preferred representatives of this group of substances consist predominantly of methylesters based on C₁₆-C₁₈ fatty acids, for example hardened beef tallow methyl esters with an average of 12 EO or an average of 20 EO.

In a preferred embodiment of the invention, a mixture is used as binder which employs C₁₂-C₁₈ fatty alcohol based on coconut or tallow with an average of 20 EO and polyethylene glycol with a molecular weight of 400 to 4000.

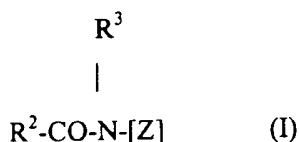
In a further preferred embodiment of the invention, a mixture is used as binder which contains predominantly methylesters based on C_{16} - C_{18} fatty acids with an average of 10 to 25 EO, in particular hardened beef tallow methyl esters with an average of 12
5 EO or an average of 20 EO and a C_{12} - C_{18} fatty alcohol based on coconut or tallow with an average of 20 EO and/or polyethylene glycol with a molecular weight of 400 to 4000.

Particularly preferred embodiments of the invention have proved to consist of binders
10 which are either based on polyethylene glycols with a molecular weight of around 4000 alone or on a mixture of C_{12} - C_{18} fatty alcohol based on coconut or tallow with an average of 20 EO and one of the fatty acid methyl ester ethoxylates described above or on a mixture of C_{12} - C_{18} fatty alcohol based on coconut or tallow with an average of
15 20 EO, one of the fatty acid methyl ester ethoxylates described above and a polyethylene glycol, in particular one with a molecular weight of around 4000.

In addition, it is possible to use, as further binder, as such or in combination with other binders, also alkyl glycosides with the general formula $RO(G)_x$ in which R represents a primary, straight-chain or methyl-branched aliphatic radical, in particular
20 one methyl- branched in position 2, with 8 to 22, preferably 12 to 18 C atoms and G is the symbol which represents a glucose unit with 5 or 6 C atoms, preferably glucose. The degree oligomerisation x which indicates the distribution of monoglycosides and oligoglycosides is any desired number between 1 and 10; preferably, x is 1.2 to 1.4. Those alkyl glycosides are suitable, in particular, which have a softening point above
25 80°C and a melting point above 140°C . Highly concentrated compounds with contents of at least 70% by weight alkyl glycosides, preferably at least 80% by weight alkyl glycosides are also suitable. By applying high shear forces, melt agglomeration and, in particular, melt extrusion can be brought about with such highly concentrated compounds even at temperatures which are above the softening point but still below
30 the melting temperature. Although alkyl glycosides can also be used as the sole binder, it is preferable to use mixtures of alkyl glycosides and other binders. In

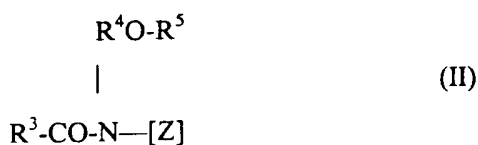
particular, mixtures of polyethylene glycol and alkyl glycosides are used here, preferably in weight ratios of 25:1 to 1:5, 10:1 to 2:1 being particularly preferred.

Polyhydroxy fatty acid amides with formula (I) are also suitable as binders, in particular in combination with polyethylene glycols and/or alkyl glycosides, in which formula R^2CO represents an aliphatic acyl radical with 6 to 22 carbon atoms, R^3 represents hydrogen, an alkyl or hydroxyalkyl radical with 1 to 4 carbon atoms and $[Z]$ represents a linear or branched polyhydroxalkyl radical with 3 to 10 carbon atoms and 3 to 10 hydroxyl groups.



Preferably, the polyhydroxy fatty acid amides are derived from reducing sugars with 5 or 6 carbon atoms, in particular from glucose.

The group of polyhydroxy fatty acid amides also includes compounds with formula (II)



in which R^3 represents a linear or branched alkyl or alkenyl radical with 7 to 12 carbon atoms, R^4 represents a linear, branched or cyclic alkyl radical or an aryl radical with 2 to 8 carbon atoms and R^5 represents a linear, branched or cyclic alkyl radical or an aryl radical or an oxy alkyl radical with 1 to 8 carbon atoms, C_1 - C_4 alkyl or phenyl radicals being preferred, and $[Z]$ represents a linear polyhydroxy alkyl radical whose alkyl chain is substituted by at least two hydroxyl groups, or alkoxyated, preferably ethoxyated or propoxyated derivatives of this radical. In this case, too, $[Z]$ is preferably obtained by the reductive amination of a sugar such as glucose, fructose, maltose, lactose, galactose, mannose or xylose. According to the theorem of the

International Patent Application WO-A-95/07331, for example, it is possible in this case to convert the N-alkoxy substituted or N-aryloxy substituted compounds by reaction with fatty acid methyl esters in the presence of an alkoxide as catalyst into the desired polyhydroxy fatty acid amides. Particularly preferred glucamides melt at temperatures of only 95 to 105°C. However, in this case, too, – as in the case of alkyl glycosides – normal operating temperatures above the softening but below the melting temperature are sufficient in the process according to the invention.

The content of binder or binders in the pre-mixture is preferably at least 2% by weight but less than 15% by weight, in particular less than 10% by weight, 3 to 6% by weight, based on the pre-mixture, being particularly preferred.

In a preferred embodiment of the process according to the invention, the solids for the production of solid flowable pre-mixture are first mixed at a temperature between room temperature and a slightly elevated temperature, preferably below the melting temperature or the softening point of the binder and in particular at temperatures of up to 35°C in a usual mixing and/or granulating device. These solids include those which, according to European Patent EP-B-0 486 592, can be used as plasticisers and/or slip additives. These include in particular anionic surfactants such as alkyl benzene sulphonates and/or (fatty) alkyl sulphates, but also polymers such as polymeric polycarboxylates. A more accurate description of the possible anionic surfactants and polymers is provided in the list of possible constituents. The function of slip agent can also be provided by the binder or the binders or the binder mixtures.

The binders are preferably admixed as the last component. As detailed above, they can be added as a solid, i.e. at a processing temperature which is below their melting point and/or their softening point, or in the form of a melt. Preferably, however, the admixing operation takes place under conditions such that as uniform and homogeneous a distribution of the binder as possible is achieved in the mixture of solids. In the case of highly finely particulate binders, this can be effected at temperatures below 40°C, e.g. at temperatures of the binder between 15 and 30°C. Preferably, however, the binder has a temperature at which it is present in the form of

a melt, i.e. above the melting point, in particular in the form of a complete melt. Preferred temperatures of the melt are between 60 and 150°C, the temperature range between 80 and 120°C being particularly preferred. During the mixing process which takes place at room temperature to slightly elevated temperatures, the melt solidifies almost instantaneously and the pre-mixture is present according to the invention in a solid, flowable form.

Joining the detergent or cleaning agent compounds and/or detergent or cleaning agent raw materials with simultaneous or subsequent forming can be effected by the usual processes in which compacting forces are applied, such as granulating, compacting, e.g. roller compacting or extruding or tableting and pelletising.

The actual granulating, compacting, tableting, pelletising or extrusion process according to the invention takes place at processing temperatures which correspond, at least in the compacting step, at least to the temperature of the softening point, or event to the temperature of the melting point. In a preferred embodiment of the invention, the process temperature is significantly above the melting point or above the temperature at which the binder is present as a melt. In particular, however, it is preferable for the process temperature in the compacting step to be not more than 20°C above the melting temperature or the upper limit of the melting range of the binder. Although it is technically altogether possible to set even higher temperatures, it has been found that a temperature difference of 20°C with respect to the melting temperature and/or the softening temperature of the binder is generally altogether sufficient and higher temperatures do not provide any additional advantages. For this reason – and in particular for energy reasons – it is particularly preferable to operate above but as closely as possible to the melting point and/or the upper temperature limit of the melting range of the binder. Such a temperature control has the further advantage that thermally sensitive raw materials such as peroxy bleaching agents such as perborate and/or percarbonate but also enzymes can also be increasingly processed without serious losses of active substance. The possibility of an accurate temperature control of the binder in particular during the decisive step of compaction, i.e. between mixing / homogenising of the pre-mixture and forming, allows a process control

which is highly advantageous from the energy point of view and extremely gentle for temperature-sensitive components of the pre-mixture since the pre-mixture is exposed to the higher temperatures only for a brief period. Preferably, the duration of the temperature exposure is between 10 seconds and maximum 5 minutes; in particular, it amounts to maximum 3 minutes.

5 In a preferred embodiment of the invention, the process according to the invention is carried out by extrusion as described in European Patent EP-B-0 486 592 or International Patent Applications WO-A-93/02176 and WO-A-94/09111 (all filed by
10 Henkel KGaA), for example. In these processes, a solid pre-mixture is compressed in the form of a strand and, after leaving the die, the strand is cut to the predeterminable granule dimension by means of a cutting device. The homogeneous, solid pre-mixture contains a plasticisers and/or slip agent which causes the pre-mixture to be
15 softened in a plastic manner under the pressure or the effect of specific operations to become extrudable. Preferred plasticisers and/or slip agents consist of surfactants and/or polymers which, within the framework of the invention as it is now present and with the exception of the above-mentioned non-ionic surfactants, are introduced into the pre-mixture not in the liquid, and in particular not in the aqueous but in the solid form.

20 To explain the extrusion process, express reference is herewith made to the above-mentioned patents and patent applications. In a preferred embodiment of the invention, the pre-mixture is in this case preferably continuously fed to a double screw extruder with synchronised or counter-current screw operation, it being
25 possible for the housing and the extruder granulation head to be heated to the predetermined extrusion temperature. Under the shearing effect of the extruder screws, the pre-mixture is compacted at a pressure, which, preferably, amounts to at least 25 bar but may be below this level at extremely high rates of throughput, depending on the equipment used, as well as plasticised, extruded through the die
30 plate in the extruder head in the form of fine strands, and the extrudate being finally comminuted by means of a rotating guillotine-type knife to form preferably approximately spherical to cylindrical granular grains. The aperture diameter of the

die and the length of the strands are chosen such as to correspond to the granule dimensions selected. In this embodiment, the production of granules with an essentially homogeneous predeterminable particle size is possible, it being possible for the individual absolute particle sizes to be adjusted to the intended application purpose. In general, particle diameters of maximum 0.8 cm are preferred. Important
5 embodiments provide here for the production of uniform granules in the mm range, for example in the range of 0.5 to 5 mm and in particular in the range of approximately 0.8 to 3 mm. The ratio of length to diameter of the cut-off primary granules in an important embodiment in this respect is within the region of
10 approximately 1:1 to approximately 3:1. Moreover, it is preferable for the still elastic primary granules to be passed to a further forming processing step; during this step, edges present on the crude extrudate are rounded off so that, in the end, spherical to near spherical extrudate grains can be obtained. If desired, small quantities of desiccant powder e.g. zeolite powder such as zeolite NaA powder can be
15 simultaneously used in this stage. Forming can be carried out in rounding devices widely available on the market. In this connection, care should be taken to ensure that only small quantities of fine granular fractions are formed in this stage. However, drying which is described in the above-mentioned documents of the state or the art as being the preferred embodiment is unnecessary within the framework of the present
20 invention since the process according to the invention is essentially free from water, i.e. is effected without the addition of free, non-combined water.

In a particularly advantageous embodiment of the invention, the binder used has a melting temperature or a melting range of up to 75°C; process temperatures which are
25 maximum 10°C and in particular maximum 5°C above the melting temperature or the upper temperature limit of the melting range of the binder have proved to be particularly advantageous.

Under these process conditions, the binder has the effect of a slip agent, in addition to
30 the above-mentioned effects, and prevents or at least reduces the occurrence of agglutinations to equipment walls and compacting tools. This applies not only to

processing in the extruder but equally to processing in continuously operating mixers/granulators or rollers, for example.

5 Immediately after leaving the manufacturing equipment, the compacted substance preferably has a temperature of not more than 60°C, temperatures between 35 and 65°C being particularly preferred. It has been found that discharge temperatures – particularly in the extrusion process – of 40 to 55°C are particularly advantageous.

10 As in the extrusion process, it is equally preferably in the other manufacturing processes to pass the primary granules/compacted substances thus formed to a further forming processing step, in particular a rounding off process, so that, finally, spherical to near spherical (bead shaped) grains can be obtained.

15 It is the essence of a preferred embodiment of the invention that the particle size distribution of the pre-mixture is considerably wider than that of the end product produced according to the invention and corresponding to the invention. In this connection, the pre-mixture can contain considerably coarser fines, and even dust fractions, if necessary also more coarse-grained fractions, it being preferable, however, for a pre-mixture with a relatively wide particle size distribution and a
20 relatively high proportion of fines to be converted into an end product with a relatively narrow particle size distribution and relatively small proportions of fines.

25 Due to the fact that the process according to the invention is carried out essentially free from water - i.e. it is free from water with the exception of the water contents ("impurities") of the solid raw materials used - not only the risk of gel formation by the surfactant raw materials in the manufacturing process itself is minimised or even eliminated, an ecologically valuable process is additionally made available since, by doing away with a subsequent drying step, it is possible not only to save energy but also to avoid emissions such as those occurring mainly in the case of conventional
30 types of drying.

According to the theorem of the above-mentioned application, it is, for example, possible to produce builder granules (builder granule extrudates), bleaching activator granules (bleach activator granule extrudates) or enzyme granules (enzyme granule extrudates), base granules, compounds and treated raw materials having a spherical or bead form being particularly preferably made available.

The process end products produced according to the theorem of the above-mentioned application have a very high bulk density. Preferably, the bulk density is above 700 g/l, in particular between 750 and 1000 g/l.

The process described in the above-mentioned earlier German patent application 196 38 599.7 is highly suitable for carrying out process step a) within the framework of the present invention. Another process preferably used as process step a) is described in the earlier German patent application 197 53 310.8 (Henkel KGaA). According to the theorem of this specification, the production of the pourable and flowable high density granules takes place in step a) by initially producing a solid pre-mixture which contains at least one non-aqueous binder and a solid raw material or solid raw materials from a raw material class, which is or are present as a solid at room temperature and a pressure of 1 bar and has or have a melting point or softening point of not less 45°C, in quantities of at least 50% by weight and converting the pre-mixture by applying compacting forces at temperatures of at least 45°C into a grain and, if necessary, processing or further treating it with the proviso that

- the pre-mixture is essentially anhydrous and
- the pre-mixture contains at least one non-aqueous binder which is present in the solid form at a pressure of 1 bar and temperatures below 45°C but is present as a melt under the processing conditions, this melt serving as a multifunctional, water-soluble binder, which melt has the function not only of a slip agent but also an adhesive function for the raw materials during the manufacture of the agent but has a disintegrating effect during the re-dissolution of the agent in an aqueous liquor,
- and a bulk density of at least 600 g/l is adjusted.

According to the theorem of this application it is possible to use as non-aqueous binders not only the binders mentioned in 196 38 599.7 but also polymers swollen in non-aqueous solution.

5 Anhydrous, swollen polymers which can also be used as binders according to the meaning of the above-mentioned application are those which lead to gel-type states in non-aqueous liquids or low water liquid mixtures (maximum water content, based on the liquid mixture: 20%). In particular, those systems of non-aqueous liquids and polymers are suitable which, at room temperature in the presence of the polymer, exhibit a viscosity at least 20 times, in particular 300 times to 5000 times that of non-
10 aqueous liquids as such. The viscosity of the binder, i.e. in this case the combination of non-aqueous liquid and polymer at room temperature is preferably in the region of 200 mPas to 10000 mPas, in particular of 400 mPas to 6000 mPas, measured by means of a Brookfield rotation viscosimeter (Brookfield DV2, spindle 2 at 20 rpm),
15 for example. At higher temperatures, for example at 60°C, the viscosity deviates preferably only relatively slightly from the values at room temperature and is preferably in the region of 250 mPas to 2500 mPas. Suitable liquids include liquid monohydric, dihydric or trihydric alcohols with boiling points (at 1 bar) above 80°C, in particular above 120°C such as, for example, n-propanol, iso-propanol, n-butanol, s-butanol, iso-butanol, ethylene glycol, 1,2 or 1,3-propylene glycol, glycerine, di or triethylene glycol or di or tripropylene glycol or their mixtures, in particular glycerine
20 and/or ethylene glycol and the representatives of the above-mentioned non-ionic surfactants which are liquid at room temperature. It is possible to add water as "swelling aid" to the organic solvent in small quantities, namely maximum 1.5% by weight, based on the end product of the swelling process. However, only so much
25 water is preferably added to the solvent that the water content of the end product is less than 1% by weight. Although it is well known that such non-ionic surfactants tend to gel on contact with water, no tackiness of the end product occurs when they are used as non-aqueous solvents for the polymer in the binder used according to the invention. Suitable polymers leading to swollen systems in such anhydrous liquids
30 are polyvinyl pyrrolidone, polyacrylic acid, co-polymers of acrylic acid and maleic acid, polyvinyl alcohol, xanthan, partly hydrolysed starches, alginates, amylopectins,

starches or celluloses carrying methylether, hydroxyethyl ether, hydroxypropyl ether and/or hydroxybutyl ether groups, phosphated starches such as starch diphosphate but also inorganic polymers such as layer silicates and their mixtures. Among the polyvinyl pyrrolidones, those with a molecular weight of maximum 30000 are preferred. Molecular weight ranges between 3000 and 30000, e.g. around 10000, are particularly preferred. The polymers which are preferably used include also hydroxypropyl starch and starch diphosphate. The concentration of the polymers in the anhydrous liquids is preferably 5% by weight to 20% by weight, in particular approximately 6% by weight to 12% by weight.

Particularly advantageous embodiments of the above-mentioned invention contain such swollen polymers as binders.

The content of binder or binders in the raw material compound described in the above-mentioned application is preferably at least 2% by weight, but less than 20% by weight, in particular less than 15% by weight, quantities in the region of 3% by weight to 10% by weight being particularly preferred.

If desired, the raw material compounds may contain in minor quantities further constituents which are solid at temperatures below 30°C (1 bars). In this respect, it is particularly desirable to choose, as further components, those which have already been mentioned above as being among the preferred raw materials and raw material classes. For example, bleach activator granules containing more than 60% by weight TAED could additionally also contain anionic surfactant or anionic surfactants, for example alkyl sulphates and/or alkyl benzene sulphonates. Since it is considered advantageous within the framework of the invention to make as high as concentration of merely one raw material available in the compound, it is preferable for the second solid raw material to be present in the compounds in quantities of maximum 30% by weight. However, apart from the first and, if necessary, also the second solid raw material, premanufactured compounds, for example surfactant compounds or spray dried powders, such as those commonly used in detergents, can also be used in addition to the actual raw material. However, the proportions of such compounds in

the compound according to the invention are preferably less than 25°C and in particular less than 20% by weight. In addition, further components such as finely particulate aluminosilicates, for example zeolite A, X and/or P, amorphous or crystalline silicates, carbonates, if necessary also sulphates in minor quantities maybe
5 present, usually in quantities not exceeding 5% by weight, based on the compound according to the invention. Finely particulate aluminosilicates, above all, can be used to powder the raw material compounds according to the invention.

10 Constituents which are liquid at temperatures below 30°C (1 bar) are not contained in the raw material compounds of the above-mentioned application – with the exception of the above-mentioned maximum 10% by weight of non-ionic surfactants and the swollen polymers used as binders.

15 It is an essential characteristic of the above-mentioned application that the raw material compounds according to the invention contain no free water, i.e. no water that is not bound to the solids in any chemical or physical form. This is possible by the essentially water-free production of the raw material compounds (compare below) as a result of which water is entrained only in quantities such as it is contained as “impurity”, so to speak, in the solid raw materials used.

20 In a preferred embodiment of the above-mentioned application, a raw material compound contains 55 to 85% by weight bleach activator, 0 to 25% by weight, preferably 5 to 22% by weight anionic surfactants such as alkyl benzene sulphonates and/or alkyl sulphates, if necessary in a pre-compounded form, 5 to 12% by weight of
25 a non-aqueous binder, preferably polyethylene glycol with a molecular weight above 3500, in particular around 4000, or of a swollen polymer.

In a further preferred embodiment of the above-mentioned application, the raw material compound additionally contains 1 to 7.5% by weight, preferably 2 to 6% by
30 weight of a non-ionic surfactant which is liquid at temperatures below 35°C (1 bar), for example a C₁₂-C₁₈ fatty alcohol with 3 to 7 EO.

In a further preferred embodiment of the above-mentioned application, the raw material compound additionally also contains 0.5 to 5% by weight of a finely particulate, in particular a non-water-soluble constituent such as an aluminosilicate as indicated above. Such components are generally used as powdering agents and can also subsequently be applied to the raw material compound, within the framework of this invention.

In a further preferred embodiment of the above-mentioned application, the raw material compound contains 60 to 85% of one or more inorganic builders, 3 to 10% by weight of a binder used according to the invention and up to 20% by weight of anionic and/or non-ionic surfactants. In particular, builder compounds are preferred which contain 50 to 65% by weight of zeolite A, X, Y and/or P and 15 to 30% by weight of an amorphous alkali silicate.

The use of anhydrous swollen polymers as slip agents and binders is also described in the earlier International Patent Application **WO/PCT/EP97/05945**. The process disclosed in this application is also suitable for the execution of step a) within the framework of the present invention. Consequently processes are also preferred within the framework of the present invention in the case of which the production of the pourable and flowable high density granules in step a) is effected by joining detergent or cleaning agent compounds and/or detergent or cleaning agent raw materials with simultaneous or subsequent forming, a pre-mixture being initially produced which contains individual raw materials and compounds present as solids at room temperature and a pressure of 1 bar and this pre-mixture being subsequently converted into a grain by the application of compaction forces and, if necessary, subsequently being further treated or processed, with the proviso that the pre-mixture is essentially anhydrous and a forming aid is used which is liquid under the forming conditions, in particular also at room temperature and a pressure of 1 bar and has the form of a polymer swollen in a non-aqueous solution.

The constituents used in the process described in this specification can consist of separately produced compounds but also of raw materials which have the form of a

powder or (finely divided to coarse) particles and, in any case, are present in the solid form at room temperature and a pressure of 1 bar – with the exception of the non-ionic surfactants liquid at temperatures below 45°C and a pressure of 1 bar, which may be present if necessary. The particulate particles used may, for example, consist of beads produced by spray drying or the agglomerates from a granulation process etc. The composition of the compounds as such is not important with respect to the invention with the exception of the water content which must be such that the pre-mixture as defined above is essentially anhydrous and preferably contains not more than 10% by weight of water of hydration and/or constitutional water. In a preferred embodiment, super-dried compounds are used in the pre-mixture. Such compounds can, for example, be obtained by spray drying, the temperature control being such that the tower discharge temperatures are above 100°C, preferably 110°C or above. It is also possible to use solid compounds in the pre-mixture which serve as carriers for liquid components, for example liquid non-ionic surfactants or silicone oil and/or paraffins. These compounds can contain water to the level indicated above, the compounds being flowable and preferably remaining flowable and/or at least conveyable at higher temperatures of at least 45°C. In particular, however, it is preferable for compounds with maximum 12% by weight and particularly preferably with maximum 9% by weight of water, based on the pre-mixture, to be used in the pre-mixture. Free water, i.e. water which not bound in any form to a solid and is consequently present “in the liquid form” should preferably not be contained in the pre-mixture since even very small quantities, for example around 0.2 or 0.5% by weight based on the pre-mixture, are sufficient to partially dissolve the forming agent which is soluble as such. The consequence would be that the melting point or the softening point of the end product would be reduced and it would lose both some of its flowability and bulk density.

Joining of the detergent or cleaning agent compounds and/or detergent or cleaning agent raw materials with simultaneous or subsequent forming can be effected according to the disclosure of the above-mentioned application by the usual processes in which compacting forces are applied such as granulation, compacting, for example roller compacting, or extruding and pelletising. It is in this connection possible to use

also spray dried granules as premanufactured compounds in the pre-mixture; however, the invention is in no way limited thereto. Instead, the process according to the invention provides for the possibility of using no spray dried granules since highly finely particulate raw materials with dust-type fractions can be processed according to the invention without problems and without being previously precompounded, e.g. spray dried.

The essentially water-free execution of the process does not only make it possible for peroxy bleaching agents to be processed without loss of activity, it is thereby also made possible to process peroxy bleaching agents and bleach activators jointly in one particle without having to fear serious losses of activity.

In a preferred embodiment of the invention of the above-mentioned application, compacting forming of the process is effected by means of an agglomeration step, the pre-mixture being subjected to agglomerating granulation in a device suitable for this purpose, with the forming agent defined above assuming the role of binder. The granulation process can be carried out continuously or batchwise. In this connection, one proceeds in such a way that the solid components of the pre-mixture to be compacted are introduced into a granulator for which a mixer can also be used, if necessary dusts which may be present being combined by the addition of a liquid non-ionic surfactants, the forming agent being introduced to the granulator. The desired average particle size of the granules can be adjusted by way of the type and quantity of forming agent and the machine and operating parameters such as the rotational speed and the residence time as well as the temperature. Suitable granulators are, for example, pelletising pan mixers, revolving drums, ploughshare mixers with choppers made by Lödige®, high performance mixers with a rotating mixer vessel and vortex device, e.g. those made by Laeis Bucher® or Eirich®, high-intensity mixers with shearing heads such as those made by LIPP Mischtechnik® or Imcaterc®, Drais®, Fukae® or Forberg® mixers as well as the so-called Rotorcoator® made by Glatt® with a horizontal rotary disc and those with a rotary disc inclined at up to 50°. Less suitable are the following: Lödige® CB mixers, zigzag mixers made by PK-Niro®,

the Ballestra® chain mix and the Hosokawa® or the Schugi® mixer. A fluid bed or a horizontal mixer, e.g. a Nautamixer® is also less suitable. Within the framework of this embodiment of the process according to the invention it is preferable to operate at room temperature or the temperature brought about by the energy input of the mixer or the granulator i.e. without a separate heating step, as described in International Patent Application WO 94/13779 and the state of the art detailed therein, for example. In this connection it should be considered as an advantage of the process according to the invention that one dose not depend on a two-stage granulating process such as that described in European Patent Application EP 0 367 339, for example, in which the granules are first compressed in a high speed mixer and subsequently in a low speed mixer and granulator, it being possible to effect the compacting granulation in a single step using the anhydrous swollen polymer.

To sum up, the first step of the process according to the invention is carried out by any usual process in which the constituents are plasticised and, by the application of shearing forces, compressed into a form. Preferred production processes are granulation, extrusion, roller compaction or pelletising. Processes which are particularly preferred operate in step a) according to the theorem of the above-mentioned earlier applications.

Process step b):

In process step b), further constituents to be optionally used are agglomerated such that they satisfy the desired selection criteria with respect to the particle size. In step b), it is not necessary to plasticise the mixture of the individual constituents to be agglomerated. Consequently, normal granulation processes are preferred for the preparation of the agglomerates in step b).

During the preparation of the agglomerates in step b) it is preferable for this also to be carried out essentially free from water, the term "essentially free from water" being understood to refer to the state as defined above in which the content of liquid water, i.e. water not present in the form of water of hydration and/or constitutional water, is

less than 5% by weight, preferably less than 3% by weight and in particular even less than 0.5% by weight, based on the substances to be granulated in step b).

5 During the granulation of the constituents to be optionally used, auxiliary agents can be added which facilitate agglomerate formation. In particular, the addition of the plasticising and/or slip agents to be used in step a) is preferable also during the granulation in step b), the mixture to be agglomerated – in contrast to step a) – containing these substances not in order to be formed by compression via the plastic state but merely as a granulating aid.

10

Preferred granulating aids in step b) are the polyethylene glycols or ethoxylated alcohols. Particularly preferably, the anhydrous swollen polymers described above are used as granulating aids.

15

In step b), it is also possible to use part of the granules obtained in step a) as base material and to "bond" further finely particulate constituents to these. Obviously, other coarser substances can be introduced as base material, more finely particulate substances being applied to them, the granulating aids having an increased significance as adhesion-promoting component.

20

Apart from the above-mentioned process-specific auxiliary agents, it is possible for all constituents of detergents and cleaning agents to be used as constituents in steps a) and b), no limits being set to the expert during formulation. It is possible, without causing any problems, to introduce individual constituents into the detergent and cleaning agent formed bodies either exclusively via process step a) or exclusively via process step b). However, it is also conceivable and possible, without causing problems, to use the constituents both in process a) and in process step b).

25

By separately incorporating certain constituents, positive effects can be achieved. For example, it is thus possible to add bleaching agents and bleach activator(s) either in process step a) or in process step b). By using bleaching agent or bleach activator in

30

step a) and the other substance in step b) it is also possible to achieve a separation of the bleaching agent and the bleach activator, leading to advantageous properties.

5 Whereas the separation of bleaching agent and bleach activator, i.e. incorporation via step a) or step b) is not preferred within the framework of the present invention, other process variants are preferred which lead to the enhanced stability of sensitive substances. In particular, it is thus preferred to add enzymes in the process step in which bleaching agent and/or bleach activator(s) are not added, in order to thus prevent a loss of activity of the enzymes. Since, moreover, enzymes are temperature-
10 sensitive substances, it is recommended to introduce them into the process according to the invention via process step b) so that it is preferable for at least one of the agglomerates prepared in step b) to contain enzymes.

15 In the following, the constituents of detergents and cleaning agents suitable for use in both step a) and in step b) will be described in further detail.

In the detergent and cleaning agent formed bodies prepared according to the process of the invention, anionic, non-ionic, cationic and/or amphoteric surfactants or mixtures of these can be used. From the point of view of technical application,
20 mixtures of anionic and non-ionic surfactants are preferred. The total surfactant content of the formed bodies produced according to the process of the invention is 5 to 60% by weight, based on the weight of the formed body, surfactant contents of more than 15% by weight being preferred.

25 The anionic surfactants used are, for example, those of the sulphonate and sulphate type. Suitable surfactants of the sulphonate type, in this respect, are preferably C₉₋₁₃ alkyl benzene sulphonates, olefin sulphonates, i.e. mixtures of alkene and hydroxyalkane sulphonates as well as disulphonates such as those obtained from C₁₂₋₁₈ monoolefins with terminal or internal double bonds by sulphonation with gaseous
30 sulphur trioxide and subsequent alkaline or acidic hydrolysis of the sulphonation products. Alkane sulphonates obtained from C₁₂₋₁₈ alkanes, e.g. by sulphochlorination or sulfoxidation with subsequent hydrolysis or neutralisation are also suitable. The

esters of alpha-sulphofatty acids (ester sulphonates), e.g. the alpha-sulphonated methyl esters of hydrogenated coconut, palm kernel or tallow fatty acids, are also suitable.

5 Other suitable anionic surfactants are the sulphonated fatty acid glycerine esters. The term fatty acid glycerine esters should be understood to refer to the monoesters, diesters and triesters as well as the mixtures thereof such as those obtained by esterification of a monoglycerine with 1 to 3 mole fatty acids or by re-esterification of triglycerides with 0.3 to 2 mole glycerine. In this respect, preferred sulphonated fatty
10 acid glycerine esters consist of the sulphonation products of saturated fatty acids with 6 to 22 carbon atoms, for example caproic acid, caprylic acid, caprinic acid, myristic acid, lauric acid, palmitic acid, stearic acid or behenic acid.

The preferred alk(ene)yl sulphates are the alkali and in particular the sodium salts of
15 the sulphuric acid half-esters of the C_{10-18} fatty alcohols, e.g. of coconut fatty alcohol, tallow fatty alcohol, lauryl, myristyl, cetyl or stearyl alcohol or the $C_{12-C_{20}}$ oxo alcohols and those half-esters of secondary alcohols having this chain length. Other preferred alk(ene)yl sulphates with the above-mentioned chain length are those which contain a synthetic straight-chain alkyl radical produced by the petrochemical route
20 which have an analogous decomposition behaviour to the adequate compounds based on fatty chemical raw materials. For wash technology reasons, the $C_{12-C_{16}}$ alkyl sulphates and $C_{12-C_{15}}$ alkyl sulphates as well as the $C_{14-C_{15}}$ alkyl sulphates are preferred. 2.3-alkyl sulphates which are produced according to US patent specifications 3,234,258 or 5,075,041, for example and can be obtained as
25 commercial products of Shell Oil Company under the tradename DAN® are also suitable anionic surfactants.

The sulphuric acid monoesters of the straight-chain or branched C_{7-21} alcohols ethoxylated with 1 to 6 mole ethylene oxide, such as 2-methyl-branched C_{9-11} alcohols
30 with, on average, 3.5 mole ethylene oxide (EO) or C_{12-18} fatty alcohols with 1 to 4 EO are also suitable. Because of their strong foaming behaviour, they are used in

cleaning agents only in relatively small quantities, e.g. in quantities of 1 to 5% by weight.

5 Further suitable anionic surfactants also consist of the salts of alkyl sulphosuccinic acid, also referred to as sulphosuccinates or as sulphosuccinic acid esters, and the monoesters and/or diesters of sulphosuccinic acid with alcohols, preferably fatty alcohols and in particular ethoxylated fatty alcohols. Preferred sulphosuccinates contain C₈₋₁₈ fatty alcohol radicals or mixtures of these. Sulphosuccinates which are particularly preferred contain a fatty alcohol radical derived from ethoxylated fatty
10 alcohols which, considered as such, represent non-ionic surfactants (for a description see below). In this respect sulphosuccinates the fatty alcohol radicals of which are derived from ethoxylated fatty alcohols with a narrow homologue distribution, are particularly preferred. It is also possible to use alk(ene)yl succinic acid with preferably 8 to 18 carbon atoms in the alk(ene)yl chain or their salts.

15 Further suitable anionic surfactants are in particular soaps. Saturated fatty acid soaps such as the salts of lauric acid, myristic acid, palmitic acid, stearic acid, hydrogenated erucic acid and behenic acid as well as soap mixtures derived in particular from natural fatty acids e.g. coconut, palm kernel or tallow fatty acids, are also suitable.

20 The anionic surfactants, including soaps, can be present in the form of their sodium, potassium or ammonium salts as well as in the form of soluble salts or organic bases such as mono, di or triethanol amine. Preferably, the anionic surfactants are present in the form of their sodium or potassium salts, in particular in the form of the sodium
25 salts.

Preferably, alkoxyated, preferably ethoxylated, in particular primary alcohols with preferably 8 to 18 C atoms and an average of 1 to 12 mole ethylene oxide (EO) per
30 mole alcohol are used as non-ionic surfactants in which the alcohol radical can be linear or preferably methyl-branched in position 2 or which may contain linear or methyl-branched radicals in the mixture in the same way as they are commonly present in oxo alcohol radicals. In particular, however, alcohol ethoxylates with

linear radicals of alcohols of native origin with 12 to 18 C atoms, e.g. of coconut, palm kernel, tallow fatty or oleyl alcohol and on average 2 to 8 EO per mole alcohol are preferred. The preferred ethoxylated alcohols include for example C₁₂₋₁₄ alcohols with 3 EO or 4 EO, C₉₋₁₁ alcohols with 7 EO, C₁₃₋₁₅ alcohols with 3 EO, 5 EO, 7 EO or 8 EO, C₁₂₋₁₈ alcohols with 3 EO, 5 EO or 7 EO and mixtures of these such as mixtures of C₁₂₋₁₄ alcohol with 3 EO and C₁₂₋₁₈ alcohol with 5 EO. The degrees of ethoxylation indicated represent statistical mean values which may consist of an integer or a fraction for a particular product. Preferred alcohol ethoxylates have a narrow homologue distribution (narrow range ethoxylates, NRE). In addition to these non-ionic surfactants, fatty alcohols with more than 12 EO can be used. Examples of these are tallow fatty alcohol with 14 EO, 25 EO, 30 EO or 40 EO.

In addition, alkyl glycosides with the general formula RO(G)_x can be used as further non-ionic surfactants, in which R represents a primary straight-chain or methyl-branched, in particular a position 2 methyl-branched, aliphatic radical with 8 to 22, preferably 12 to 18 C atoms and G is the symbol which represents a glucose unit with 5 or 6 C atoms, preferably glucose. The degree of oligomerisation x which indicates the distribution of monoglycosides and oligoglycosides is any number between 1 and 10; preferably x is 1.2 to 1.4.

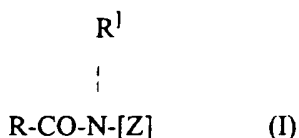
A further group of non-ionic surfactants, which are preferably used and are used either as sole non-ionic surfactant or in combination with other non-ionic surfactants, are the alkoxylated, preferably ethoxylated or ethoxylated and propoxylated fatty acid alkyl esters, preferably with 1 to 4 carbon atoms in the alkyl chain, in particular fatty acid methyl esters such as those described in Japanese patent application JP 58/217598, for example, or which are preferably prepared according to the process described in International Patent Application WO-A-90/13533.

Non-ionic surfactants of the amine oxide type, for example N-coconut alkyl-N,N-dimethyl amine oxide and N-tallow alkyl-N,N-dihydroxyethyl amine oxide and of the fatty acid alkanol amide type may also be suitable. The quantity of these non-ionic

surfactants is preferably not more than that of the ethoxylated fatty alcohols, in particular not more than half of these.

Other suitable surfactants are the polyhydroxy fatty acid amides of formula (I)

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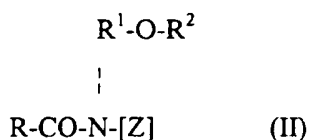
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in which RCO represents an aliphatic acyl radical with 6 to 22 carbon atoms, R^1 represents hydrogen, an alkyl or hydroxy alkyl radical with 1 to 4 carbon atoms and a [Z] represents a linear or branched polyhydroxy alkyl radical with 3 to 10 carbon atoms and 3 to 10 hydroxyl groups. The polyhydroxy fatty acid amines are known substances which can common be obtained by the reductive amination of a reducing sugar with ammonia, an alkyl amine or an alkanol amine and subsequent acylation with a fatty acid, a fatty acid alkyl ester or a fatty acid chloride.

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The group of polyhydroxy fatty acid amides includes also compounds of formula (II),

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in which R represents a linear or branched alkyl or alkenyl radical with 7 to 12 carbon atoms, R^1 represents a linear, branched or cyclic alkyl radical or an aryl radical with 2 to 8 carbon atoms and R^2 represents a linear, branched or cyclic alkyl radical or an aryl radical or an oxy alkyl radical with 1 to 8 carbon atoms, C_{1-4} -alkyl or phenyl radicals being preferred and [Z] represents a linear polyhydroxy alkyl radical whose alkyl chain is substituted by at least two hydroxyl groups, or alkoxyated, preferably ethoxylated or propoxylated derivates of this radical.

30

[Z] is preferably obtained by the reductive amination of a reducing sugar, e.g. glucose, fructose, maltose, lactose, galactose, manose or xylose. The N-alkoxy or N-aryloxy-substituted compounds can then, for example, be converted into the desired

polyhydroxy fatty acid amides according to the theorem of the international application WO-A-95/07331 by conversion with fatty acid methyl esters in the presence of an alkoxide used as catalyst.

5 Within the framework of the present invention, it is preferable to introduce anionic and non-ionic surfactant(s) via the process into the detergent and cleaning agent formed bodies, the advantages in terms of technical application being obtained from certain quantitative ratios in which the individual surfactant classed can be used.

10 Thus, detergent and cleaning agent formed bodies are, for example, particularly preferred in the case of which the ratio of anionic surfactant(s) to non-ionic surfactant(s) is between 10:1 and 1:10, preferably between 7.5:1 and 1:5 and in particular between 5:1 and 1:2, it being possible to introduce the surfactants in the process according to the invention into the formed bodies again via steps a) and/or b).

15 From the technical applications point of view, it may be advantageous if certain surfactant classes are not present in some phases of the detergent and cleaning agent formed bodies or in the overall formed bodies, i.e. in all phases. The term phase within the framework of the present invention should be understood to mean any
20 spatial separation, i.e. for example, the multiple phase state in multiple layer or ring/nucleus or jacketed tablets. In this connection, individual phases are also formed by the coarse particles from steps a) and b) of the process according to the invention being compressed, the particles from process step a) forming a phase whereas the other phase is formed by the particles from process step b). It is also quite
25 conceivable to produce detergent and cleaning agent formed bodies containing four different phases by preparing, mixing and tableting a granular product from step a) and three granular products from step b) or visa versa or to produce, mix and tablet two granular products from steps a) and b) each. This tableting process may additionally lead to multiple layer formed bodies, if this is desired.

30 A further important embodiment of the present invention provides for at least one phase of the formed bodies to be free from non-ionic surfactants. This variant can be

achieved particularly easily by the process according to the invention if non-ionic surfactants from one of process steps a) or b) are completely omitted.

5 Conversely however, a positive effect can also be achieved by the content of certain surfactants in the individual phases or the total formed body, i.e. all phases,. The introduction of the alkyl polyglycosides described has proved advantageous in this connection so that detergent and cleaning agent formed bodies are preferred in which at least one phase of the formed body contains alkyl polyglycosides; this can again be achieved by introducing APG into process step a) and/or b).

10 Similar to the case of non-ionic surfactants, it also possible, by omitting anionic surfactants from individual or all the phases, to obtain detergent and cleaning agent formed bodies which are better suited for certain fields of application. Within the framework of the present invention, detergent and cleaning agent formed bodies are
15 therefore also conceivable in which at least one phase of the formed body is free from anionic surfactants, this possible implementation being particularly easy to realise by the process according to the invention in a manner analogous to the above, if anionic surfactants from process step a) or b) are completely omitted.

20 Apart from surfactant substances, builders are the most important constituents of detergents and cleaning agents. By way of the process according to the invention, any builders commonly used in detergents and cleaning agents can be introduced into the detergent and cleaning agent formed bodies, in particular zeolites, silicates, carbonates, organic co-builders and – if no ecological prejudices exist against their
25 use – also the phosphates.

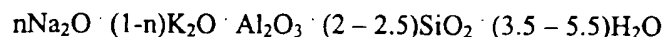
 Suitable crystalline, layer-type sodium silicates have the general formula $\text{NaMSi}_x\text{O}_{2x+1}\text{H}_2\text{O}$, M representing sodium or hydrogen, x being a number between 1.9 and 4 and y and number between 0 and 20, the preferred value for x being 2, 3 or 4.
30 Such crystalline layer silicates are described in European Patent Application **EP-A-0 164 514**, for example. Preferred crystalline layer silicates of the formula indicated above are those in which M represents sodium and x assumes the values 2 or 3. In

particular, both β and δ -sodium disilicates $\text{Na}_2\text{Si}_2\text{O}_5\text{yH}_2\text{O}$ are preferred, β -sodium disilicate being, for example, obtainable according to the process described in International Patent Application **WO-A-91/08171**.

5 Amorphous sodium silicates with a modulus of $\text{Na}_2\text{O} : \text{SiO}_2$ of 1:2 to 1:3.3, preferably 1:2 to 1:2.8 and in particular 1:2 to 1:2.6 which have a delayed dissolution and secondary wash properties can also be used. The delayed dissolution compared with conventional amorphous sodium silicates may have been brought about in different ways, e.g. by surface treatment, compounding, compacting/compression or by super-
10 drying. Within the framework of this invention, the term "amorphous" can also be understood to mean "X-ray amorphous". This means that in X-ray diffraction experiments, the silicates provide no clear-cut X-ray reflexes such as those typical of crystalline substances but at most one or several peaks of scattered X-ray radiation having a width of several degree units of the diffraction angle. However, it is
15 altogether possible to obtain particularly good builder properties if the silicate particles provide indistinct or even clear diffraction peaks during electron diffraction experiments. This should be interpreted to mean that the products exhibit microcrystalline areas of a size of 10 to some 100 nm, values of up to maximum 50 nm and in particular of up to maximum 20 nm being preferred. These so-called X-ray
20 amorphous silicates which also exhibit a delayed dissolution compared with the conventional waterglass products, are described in German Patent Application **DE-A-44 00 024**, for example. Compressed/compacted amorphous silicates, compounded amorphous silicates and super-dried X-ray amorphous silicates are particularly preferred.

25 The finely crystalline synthetic zeolite containing bound water consists preferably of zeolite A and/or P. The particularly preferred zeolite P is zeolite MAP® (commercial product from Crosfield). However, zeolite X and mixtures of A, X and/or P are also suitable. Commercially available and to be preferably used within the framework of
30 the present invention is, for example, also a co-crystalisate of zeolite X and zeolite A (approximately 80% by weight zeolite X) which is being marketed by CONDEA

August S.p.A. under the tradename VEGOBOND AX® and can be described by the formula



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The zeolite can be used both as a builder in a granular compound and as a type of "powdering agent" on the total mixture to be compressed, usually both methods of incorporation of the zeolite into the pre-mixture being used. Suitable zeolites have an average particle size of less 10 μm (volume distribution; method of measurement: Coulter Counter) and preferably contain 18 to 22% by weight, in particular 20 to 22% by weight of combined water.

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Obviously, using the generally known phosphates as builders is also possible, provided such a use ought not to be avoided for ecological reasons. In particular, the sodium salts of the orthophosphates, pyrophosphates and in particular the tripolyphosphates are suitable.

15

Suitable organic builders are, for example, the polycarboxylic acids that can be used in the form of their sodium salts such as citric acid, adipic acid, succinic acid, glutaric acid, tartaric acid, sugar acids, amino carboxylic acids, nitrilotriacetic acid (NTA) provided there is no objection to their use for ecological reasons, as well as mixtures thereof. Preferred salts consist of the salts of the polycarboxylic acids such as citric acid, adipic acid, succinic acid, glutaric acid, tartaric acid, sugar acids and mixtures thereof.

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In order to facilitate the breaking down of highly compacted formed bodies, it is possible to incorporate disintegration aids, the so-called tablet disintegration promoters into them in order to shorten the time required for breaking down. According to Römpp (edition 9, volume 6, page 4440) and Voigt "*Lehrbuch der pharmazeutischen Technologie*" (edition 6, 1987, page 182-184), the term tablet disintegration promoters or breaking down accelerators should be understood to mean

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auxiliary agents which ensure the rapid breaking down of tablets in water or gastric juice and the liberation of pharmaceuticals in a resorbable form.

5 These substances which, because of their effect, are also referred to as "disintegrating agents" increase their volume on access of water, their inherent volume being increased on the one hand (swelling) and a pressure being possibly produced, on the other hand, by the liberation of gasses, which pressure causes the tablet to break down into smaller particles. Known disintegration aids are, for example the (hydrogen-)carbonate/citric acid systems, although other organic acids can be used. For
10 example, synthetic polymers such as polyvinylpyrrolidone (PVP) or natural polymers or modified natural substances such as cellulose and starch and their derivatives, alginates or casein derivatives, for example, are swelling disintegration aids.

15 Preferred detergent and cleaning agents formed bodies contain 0.5 to 10% by weight, preferably 1 to 8% by weight and in particular 2 to 6% by weight of a disintegration aid, based on the weight of the formed body.

As preferred disintegration aids within the framework of the present invention, disintegration aids based on cellulose are used so that preferred detergent and cleaning
20 agent formed bodies contain such a cellulose-based disintegration aid in quantities of 0.5 to 10% by weight, preferably 1 to 8% by weight and in particular 2 to 6% by weight. Pure cellulose has the formal gross composition $(C_6H_{10}O_5)_n$ and, formally considered, represents a β -1,4-polyacetal of cellobiose which, in turn, is built of two
25 molecules of glucose. Suitable celluloses in this respect consist of approximately 500 to 5000 glucose units and consequently have a molecular weight of 50000 to 500000. Within the framework of the present invention, cellulose derivatives can also be used as cellulose-based disintegration aids which are obtainable from cellulose by polymer-analogous reactions. Such chemically modified celluloses comprise
30 products from esterification or etherification reactions in which hydroxy hydrogen atoms were substituted. However, celluloses in which the hydroxy groups were replaced by functional groups not bound via an oxygen atom can be used as cellulose

derivatives. For example, alkali celluloses, carboxymethyl cellulose (CMC), cellulose ester and ether as well as amino celluloses belong to the group of cellulose derivatives.

5 The above-mentioned cellulose derivatives are preferably used as cellulose-based disintegration aids not as such but in mixture with cellulose. The content of cellulose derivatives in this mixture is preferably below 50% by weight, particularly preferably below 20% by weight, based on the cellulose disintegration aid. Particularly preferably, pure cellulose free from cellulose derivatives is used as cellulose-based disintegration aid.

10

Microcrystalline cellulose can be used as a further cellulose-based disintegration aid or as part of this component. This microcrystalline cellulose is obtained by partial hydrolysis of celluloses under conditions which attack and completely dissolve only the amorphous regions (approximately 30% of the total cellulose mass) but leave the crystalline areas (approximately 70%) undamaged. A subsequent deaggregation of the micro-fine celluloses formed by hydrolysis provides microcrystalline celluloses which exhibit primary particle sizes of approximately 5 μm and can be compacted to form granules with an average particle size of 200 μm .

15

20 Within the framework of the present invention, detergent and cleaning agent formed bodies are preferred which additionally contain a cellulose-based disintegration aid in the formed bodies. Particularly preferred cellulose-based disintegration aids are agglomerated by compacting and water-free agglomeration processes and consist in a proportion of at least 90% by weight of particles with sizes above 400 μm , preferably in a proportion of at least 66% by weight of particles with sizes above 800 μm and in particular in a proportion of at least 50% by weight of particles with sizes above 1200 μm .

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30 Before the preparation of the pre-mixture (process step c)) the granules or agglomerates from process steps a) and b) can be "powdered" with finely particulate surface treatment agents. This may be advantageous for the quality and physical

properties of the pre-mixture (storage, compression) and that of the finished detergent and cleaning agent formed bodies. Finely divided powdering agents are well known in the state of the art, zeolites, silicates and other inorganic salts being generally used. Preferably, however, the pre-mixture is "powdered" with finely particulate zeolite, zeolite of the faujasite type being preferred.

Within the framework of the present invention it is preferable for at least one of the process end products from steps a) and b) to be subsequently powdered with substances in powder form, the powdering agent being a zeolite of the faujasite type with particle sizes below $100\text{ }\mu\text{m}$, preferably below $10\text{ }\mu\text{m}$ and particularly below $5\text{ }\mu\text{m}$, at least 0.2% by weight, preferably at least 0.5% by weight and in particular more than 1% by weight being used. A precondition for powdering is that the powdering agent is applied onto the process end products from steps a) or b) without detaching itself, i.e. does not lead to undesirable fines present within the framework of the present invention.

Apart from the above-mentioned components, namely surfactant, builder and disintegration aid, other constituents common in detergents and cleaning agents belonging to the group of bleaching agents, bleach activators, enzymes, fragrances, perfume carriers, fluorescent agents, dyes, foam inhibitors, silicone oils, anti-redeposition agents, optical brighteners, greying inhibitors, colour transfer inhibitors and corrosion inhibitors can be introduced via the process according to the invention into the detergent and cleaning agent formed bodies.

Among the compounds used as bleaching agents and supplying H_2O_2 in water, sodium perborate tetrahydrate and sodium perborate monohydrate have particular significance. Other suitable bleaching agents are, for example, sodium percarbonate, peroxyphosphates, citrate perhydrate-supplying and H_2O_2 -supplying peracid salts or peracids such as perbenzoates, peroxophthalates, diperazelaic acid, phthaloimino peracid or diperdodecane dioic acid.

To achieve an improved bleaching effect during washing at temperatures of 60°C and less, bleach activators can be incorporated as the only component or as a constituent of component b). Compounds providing aliphatic peroxycarboxylic acids with preferably 1 to 10 C atoms, in particular 2 to 4 C atoms and/or if necessary substituted perbenzoic acids under perhydrolysis conditions can be used as bleach activators. Suitable substances are those carrying the O and/or N acyl groups with the above-mentioned number of C atoms and/or, if necessary substituted, benzoyl groups. Multiply acylated alkylene diamines, in particular tetraacetylenediamine (TAED), acylated triazine derivatives, in particular 1,5-diacetyl-2,4-dioxohexahydro-1,3,5-triazine (DADHT), acylated glycolurils, in particular tetraacetyl glycoluril (TAGU), N-acyl imides, in particular N-nonanoyl succinimide (NOSI), acylated phenol sulphonates, in particular n-nonanoyl or isononanoyl oxybenzene sulphonate (n or iso-NOBS), carboxylic anhydrides, in particular phthalic anhydride, acylated polyhydric alcohols, in particular triacetin, ethylene glycol diacetate and 2,5-diacetoxy-2,5-dihydrofuran are preferred.

Apart from or instead of the conventional bleach activators, so-called bleach catalysts can also be incorporated into the formed bodies. These substances are bleaching effect enhancing transition metal salts or transition metal complexes such as, for example, Mn, Fe, Co, Ru or Mo salt complexes or carbonyl complexes. Mn, Fe, Co, Ru, Mo, Ti, V and Cu complexes with N-containing tripod ligands and Co, Fe, Cu and Ru ammine complexes can also be used as bleach catalysts.

Suitable enzymes are those from the class of proteases, lipases, amylases, cellulases or their mixtures. Particularly suitable are enzymatic active agents obtained from bacteria strains or fungi such as *Bacillus subtilis*, *Bacillus licheniformis* and *Streptomyces griseus*. Preferably, proteases of the subtilisin type and, in particular, proteases obtained from *Bacillus lentus* are used. Enzyme mixtures, for example those of protease and amylase or protease and lipase or protease and cellulase or cellulase and lipase or of protease, amylase and lipase or protease, lipase and cellulase, in particular, however, cellulase-containing mixtures, are of particular interest. Peroxidases and oxidases have proved suitable in some cases. The enzymes

can be adsorbed to carrier substances and/or embedded in coating substances in order to protect them against premature decomposition. The proportion of enzymes, enzyme mixtures or enzyme granules in the formed bodies according to the invention can, for example, be approximately 0.1 to 10% by weight, preferably 0.5 to approximately 5% by weight.

In addition, the detergent and cleaning agent formed bodies can also contain components which have a positive influence on the removal of oil and fats from textiles by washing (so-called soil repellents). This effect becomes particularly obvious in cases where a textile is soiled which had previously been repeatedly washed with a detergent according to the invention containing this oil and fat removing component. The preferred oil and fat removing components include, for example, non-ionic cellulose ethers such as methyl cellulose and methyl hydroxypropyl cellulose with a proportion of methoxyl groups of 15 to 30% by weight and of hydroxypropoxyl groups of 1 to 15% by weight, based on the non-ionic cellulose ether in each case, and the polymers, known from the state of the art, of phthalic acid and/or terephthalic acid or of their derivatives, in particular polymers of ethylene terephthalates and/or polyethylene glycol terephthalates or anionic and/or non-ionic modified derivatives thereof. Among these, the sulphonated derivatives of the phthalic acid polymers and terephthalic acid polymers are particularly preferred.

The formed bodies may contain derivatives of diamino stilbene disulphonic acid or their alkali metal salts as optical brighteners. Suitable salts are e.g. the salts of 4,4'-bis(s-anilino-4-morpholino-1,3,5-triazinyl-6-amino)stilbene-2,2'-disulphonic acid or similarly structured compounds which, instead of the morpholino group, carry a diethanol amino group, a methyl amino group, an anilino group or a 2-methoxyethyl amino group. In addition, brighteners of the type of the substituted diphenyl styryls can be present, e.g. the alkali salts of 4,4'-bis(2-sulphostyryl)-diphenyl, 4,4'-bis(4-chloro-3-sulphostyryl)-diphenyl or 4-(4-chlorostyryl)-4'-(2-sulphostyryl)-diphenyl. Mixtures of the above-mentioned brighteners can also be used.

Dyes and perfumes are added to agents according to the invention in order to improve the aesthetic impression of the products and to provide the user not only with softness performance but also with a product that is "typical and non confusable" from the visual and sensory point of view. Suitable perfume oils or fragrances may consist of individual odoriferous compounds, e.g. synthetic products of the ester, ether, aldehyde, ketone, alcohol and hydrocarbon type. Odoriferous compounds of the ester type are, for example: benzyl acetate, phenoxyethyl isobutyrate, p-tert.butyl cyclohexyl acetate, linalyl acetate, dimethyl benzylcarbinyl acetate, phenylethyl acetate, linalyl benzoate, benzyl formate, ethyl methyl phenyl glycinate, allyl cyclohexyl propionate, styrallyl propionate and benzyl salicylate. The ethers include, for example, benzyl ethyl ether, the aldehydes include, for example, the linear alkanals with 8 - 18 C atoms, citral, citronellal, citronellyl oxyacetaldehyde, cyclamen aldehyde, hydroxycitronellal, lilial and bourgeonal, the ketones, for example, include ionones, α -isomethyl ionone and methyl-cedryl ketones, the alcohols include anethol, citronellol, eugenol, geraniol, linalool, phenyl ethyl alcohols and terpineol, the hydrocarbons include mainly the terpenes such as limonene and pinene. Preferably, however, mixtures of different odoriferous substances are used which jointly produce an agreeable fragrance. Such perfume oils may also contain mixtures of natural odoriferous substances such as those obtainable from plant sources, e.g. pine, citrus, jasmine, patchouli, rose or ylang ylang oil. Mace, sage oil, camomile oil, clove oil, melissa oil, mint oil, cinnamon leaf oil, lime blossom oil, elderberry oil, vetiver oil, olibanum oil, galbanum oil and labdanum oil as well as orange blossom oil, neroli oil, orange peel oil and sandalwood oil are also suitable.

The fragrances can be incorporated directly into the agents according to the invention; however, it may also be advantageous to apply the fragrances onto carriers which enhance the adhesion of the perfume to the clothes and ensure a lasting fragrance of the textiles by slow fragrance release. Cyclodextrines, for example, have proved suitable for use as such carrier materials, it being possible to additionally coat the cyclodextrine perfume complexes with further auxiliary agents.

Within the framework of the present invention, it is particularly preferred to incorporate the fragrances via concentrated fragrance compounds, the so-called "fragrance beads" into the detergent and cleaning agent formed bodies. Such concentrated fragrance compounds can be produced according to process steps a) or b), it being preferable for at least one of the agglomerates produced in process step b) to contain perfume.

The production of the "fragrance beads" preferably used in the process according to the invention is described in the earlier German Patent Application 197 46 780.6 (Henkel KGaA), for example. This application discloses a process for the production of fragrance formed bodies, in particular fragrance beads with bulk densities above 700 g/l, in the case of which a solid and essentially anhydrous pre-mixture of

- a) 65 to 95% by weight carrier substance(s)
- b) 0 to 10% by weight auxiliary agent(s) and
- c) 5 to 25% by weight perfume

is subjected to granulation or compression agglomeration.

In this connection, the preferred carrier substances are selected from the group of surfactants, surfactant compounds, di and polysaccharides, silicates, zeolites, carbonates, sulphates and citrates and are used in quantities of between 65 and 95% by weight, preferably of between 70 and 90% by weight, based on the weight of the fragrance formed bodies produced.

Apart from the "fragrance beads" that can be prepared by way of the process described above, the incorporation of fragrance beads as described in the earlier German Patent Application 197 46 781.4 (Henkel KGaA) is preferred in the process in the present invention. In this specification, a process for the production of fragrance-enhanced detergents or cleaning agents or components for these with bulk densities above 600 g/l is disclosed, in which a solid and essentially anhydrous pre-mixture of detergent or cleaning agent compounds and/or detergent or cleaning agent raw materials is produced which contains at least 0.1% by weight perfume, based on the pre-mixture, and this pre-mixture is subjected to compression agglomeration.

Such fragrance-enhanced detergents or cleaning agents or the above-mentioned fragrance formed bodies can be used as the end products from a process step a) or b) also in the process according to the invention, provided they satisfy the selection criteria with respect to particle size.

5

In order to improve the aesthetic impression of the agents according to the invention, they can be dyed with suitable dyes. Preferred dyes, the selection of which causes no problems whatsoever to the expert, have a high stability in storage and insensitivity vis-à-vis the other constituents of the agents and vis-à-vis light as well as no pronounced substantivity vis-à-vis textile fibres, so as not to discolour the latter. -

10

For aesthetic reasons it may be desirable to produce formed bodies in which only individual phases or layers are dyed. In the process according to the invention, this is possible without problems in the different variations. Thus - due to the process steps a) and b) - at least two phases are already present in the formed body only one or even both of which can be dyed differently, thus obtaining "mottled" formed bodies.

15

Dyeing of the process end products from intermediate steps a) or b) can be effected in the conventional way by the addition of dyes or dye solutions. However, the use of fully dyed powder agents is preferred which cover the surface of the process end products from the intermediate steps and thus optically suggest a fully dyed grain. In this way, dye is saved, on the one hand, and, on the other hand, problems due to the incorporation of excessive quantities of dye into the formed bodies and consequently into the wash liquor are avoided.

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Process step c):

Process c) comprises combining the granules from steps a) and b) to form a compressed pre-mixture. In the case of multiple layer tablets, several differently composed and/or dyed pre-mixtures are provided. As already explained in terms of an approach, it is not necessary in this respect to homogeneously mix the process end products from intermediate steps a) and b), this procedure being preferred. According to the invention, it is also possible to produce double and multiple layer tablets by

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combining the granules from steps a) and b) only immediately before process step d), namely the compressing step.

5 However, since multiple layer or ring/nucleus or jacketed tablets require an increased process effort and reduce the rates of throughput (number of formed bodies per unit of time) in the tableting press, it is preferable within the framework of the present invention to mix the process end products from intermediate steps a) and b) before introduction into the matrix. Mixing of the process end products from intermediate steps a) and b) can appropriately be effected in the mixer which was used for the
10 preparation of the agglomerates in step b). A batchwise process would be such that intermediate steps a) and b) would be carried out in parallel and the process end products from step a) transferred to the mixer used in step b) where they would be mixed with the process end products from step b). The pre-mixtures produced in this way could then undergo intermittent storage until processed further in step d).

15 However, it is also possible to design a continuous process in the case of which step c) is carried out in a further mixer from which the pre-mixture to be compressed is then passed to a tableting press. This process variant is required in particular in those cases where, in step b) not just one granular product is formed but several differently
20 composed granular products all of which are to be combined in step c).

When selecting suitable machines and process parameters, the expert is able to fall back on machines and equipment known in the literature and on technical process operations such as those described by *W. Pietsch, in "Size Enlargement by Agglomeration", publisher: Wiley, 1991* and the literature quoted therein.
25

The proportion of the individual process end products from steps a) and b) in the pre-mixture can vary within wide limits, depending on which type of formed body is to be produced. It is preferable during the production of the pre-mixture for the portion of the granules produced in step a) present in pre-mixture c) to amount to 40 to 95% by weight, preferably 50 to 90% by weight and in particular 60 to 85% by weight, based on the pre-mixture.
30

Process step d):

Process step d) is executed by filling the pre-mixture produced in step c) into the appropriate matrices and forming it, in particular compressing it to tablets or pellets, it
5 being possible to fall back on conventional processes for the production of formed bodies. In the tableting press, the pre-mixtures are compacted in a so-called matrix between dies to form a firm compressed body. This process, referred to simply as tableting in the following, consists of four stages, metering, compaction (elastic deformation), plastic deformation and ejection.

10 Tableting takes place in commercially available tableting presses which, basically, may be equipped with single or double dies. In the latter case, the upper die is not only used for a pressure build up, the lower die moves during the compression process towards the upper die while the upper die presses downwards. For small production
15 quantities, eccentric tableting presses are preferably used in the case of which the die or dies are fixed to an eccentric disc which in turn is mounted to a shaft with a certain rate of rotation. The movement of this compression die is comparable to the method of operation of a standard four-stroke engine. Compression can be effected with any upper and lower die; however, several dies can also be fixed to an eccentric disc, the
20 number of matrix bores being correspondingly increased. The rates of throughput of the eccentric presses vary, depending on the type, between several hundred and maximum 3000 tablets per hour.

For higher rates of throughput, rotary tableting presses are chosen in the case of which
25 a larger number of matrices is arranged in a circular manner on a so-called matrix table. The number of matrices varies between 6 and 55, depending on the model, larger matrices also being available on the market. An upper and a lower die is allocated to each matrix on the matrix table, it being again possible to build up the compression pressure actively only via the upper or the lower die but also by both
30 dies. The matrix table and the dies move jointly around a vertical shaft, the dies being placed by means of rail-type curved paths during rotation into the positions for filling, compaction, plastic deformation and ejection. At those points where a particularly

pronounced lifting or dropping of the dies is required (filling, compaction, ejection), these curved paths are supported by additional low pressure pieces, low traction rails and ejection paths. Filling of the matrices takes place via a rigidly arranged input device, the so-called filling shoe, which is connected to a storage container for the pre-mixtures. The compression pressure onto the pre-mixture concerned is individually adjustable via the upper and lower die compression paths, the pressure build up being effected by rolling the die shaft heads past adjustable pressure rollers. To increase the rate of throughput, the rotary presses can also be equipped with two or several filling shoes. To produce double or multiple layer formed bodies, several filler shoes are arranged behind each other without the slightly pressed down first layer being ejected before further filling. By way of a suitable process control, jacketed and point type tablets can be produced which have an onion skin type structure, the upper side of the nucleus or the nucleus layers not being covered in the case of the point type tablets, thus remaining visible. The rotary tableting presses can also be equipped with single or multiple tools so that, for example, an outer circle with 50 and an inner circle with 35 bores can be used simultaneously for compressing. The rates of throughput of modern rotary tableting presses are one million formed bodies per hour.

Tableting machines which are suitable within the framework of the present invention are, for example supplied by Apparatebau Holzwarth GbR, Asperg, Wilhelm Fette GmbH, Schwarzenbek, Hofer GmbH, Weil, KILIAN, Cologne, KOMAGE, Kell am See, KORSCH Pressen GmbH, Berlin, Mapag Maschinenbau AG, Bern (CH) and Courtoy N.V., Halle (BE/LU). The hydraulic double pressure press HPF 630 manufactured by LAEIS, for example, is particularly suitable.

The formed bodies can be manufactured in a predetermined dimensional form and predetermined size, it being possible for them to consist of several phases, i.e. layers, inclusions or nuclei and rings. Suitable dimensional forms are, in practice, all manageable designs e.g. shaping them as a tablet, stick, rod or bar, cube, rectangular block and corresponding spatial elements with level lateral surfaces as well as, in particular, cylindrical designs with a circular or oval cross-section. This latter design

includes the provision of forms ranging from the tablet to the compact cylinder with a ratio of height to diameter of more than 1.

5 The apportioned compacted bodies can, in each case, be shaped in the form of separate individual elements which correspond to the predetermined dosage of the detergent and/or cleaning agent. It is also possible to form compressed bodies combining a number of such mass units in one compressed body, the easy separability of apportioned smaller units being provided in particular by predetermined breaking points. For the use of textile detergents in machines of the type common in Europe
10 with horizontal mechanics, the formation of the apportioned compressed bodies as tablets, in cylinder or cube form may be appropriate, a ratio of diameter to height in the region of approximately 0.5 : 2 to 2 : 0.5 being preferable. Commercial hydraulic presses, eccentric presses or rotary presses, in particular, are suitable devices for the production of such compressed bodies.

15 The dimensional form of another embodiment of the formed body has been adjusted, in terms of its dimensions, to the dispenser chamber of commercial domestic washing machines so that the formed body can be metered directly without metering aid into the dispenser chamber where it dissolves during the flushing-in process. As a matter
20 of course, however, the use of the detergent formed bodies via a metering aid is also possible without problems.

A further preferred formed body, which can be produced, has a plate or board-type structure with alternate thick and long and thin and short segments so that individual
25 segments can be broken off from the "bar" at the rated breaking points representing the short thin segments and introduced into the machine. This principle of "bar-shaped" formed body detergent can also be realised in the form of other geometric shapes, e.g. vertical upright triangles which are merely joined along one of their sides longitudinally. In this case, the possibility presents itself for optical reasons to form
30 the triangular base which connects the individual segments as one phase while the tip of the triangle forms the second phase. Dyeing the two phases differently is particularly attractive in the case of this embodiment.

Following compression, the detergent and cleaning agent formed bodies exhibit a high level of stability. The fracture strength of cylindrical formed bodies can be determined by way of the measured value of the diametral fracture stress. This can be
 5 determined according to the following formula

$$\sigma = \frac{2P}{\pi Dt}$$

10 in which σ represents the diametral fracture stress, DFS, in Pa, P is the force in N which leads to the pressure exerted onto formed body causing the fracture of the formed body, D is the formed body diameter in meters and t is the height of the formed body.

15 According to the process of the invention it is possible and preferred to adjust compression pressures in process step d) which lead to formed bodies with fracture hardness of 20 to 150 N, preferably of 40 to 100 N and in particular of 50 to 80 N. If multiple phase detergent and cleaning agent formed bodies are produced by the process of the invention, it is again preferable for the fracture hardness of the
 20 individual phases to differ by maximum $\pm 15\%$, preferably by maximum $\pm 10\%$.

By way of the quantity of pre-mixture compressed in process step d), the mass of the formed body can be accurately fixed. Within the framework of the present invention, processes are preferred which lead to detergent and cleaning agent formed bodies with
 25 a weight of between 10 and 150 g, preferably between 20 and 100 g and in particular between 35 and 75 g.

The process according to the invention is particularly suitable for the production of universal detergents in tablet form. However, colour care detergents can also be
 30 produced by omitting the use of bleaching agents and bleach activators and instead introducing coarse-grained salts or organic oligocarboxylic acids, e.g. sodium citrate or citric acid into the formed bodies. Special detergents in the form of compact formed bodies can also be produced without problems by adding textile care

substances, for example, in one of the process steps a) or b) or matching certain constituents to the required profile for certain textiles. According to the process of the invention, it is thus also possible without problems to provide wool detergents in tablet form, for example. The expert is not restricted in his formulation freedom by the process of the invention and is now able to produce, by way of the process according to the invention, standard, universal and special detergents familiar to him also in the form of compacted formed bodies. Examples of formulations for a wide variety of detergents are given in W.H. de Groot, I. Adamai, G.F. Moretti, *"The Manufacture of Modern Detergent Powders"*, published by W. Hermann de Groot Academic Publishers, 1995, for example.

The process according to the invention has a number of advantages compared with conventional production processes for detergent and cleaning agent formed bodies. On the one hand, the particle size of the pre-mixture to be compressed is substantially greater than normally, on the other hand, the particle size distribution is relatively narrow. Without wanting to be restricted by the theory, the applicant starts out from the assumption that it is possible, as a result of the larger particles, to exert a higher compression pressure onto the mixture to be tableted without negatively influencing the solubility thereof. In the case of conventional more finely particulate pre-mixtures, compaction is brought about by the compression pressure in that the fine particles fill the gaps between the coarser particles leading, in extreme cases, to a compact though firmly bonded formed body throughout which is difficult to dissolve. By almost completely eliminating finely particulate substances from the pre-mixture and by way of a relatively narrow size distribution, formed bodies can be produced by the process according to the invention which exhibit a void between the firmly bonded "beads" and are easily soluble in spite of their high density. This effect is enhanced by using large proportions of process end products from intermediate step a).

Due to the excellent reproducibility during the production of the process end products in intermediate step a), the pre-mixture has a highly homogeneous composition. Each (ideally "bead shaped") granule grain from intermediate step a) exhibits, due to the

process, the same composition and always almost the same size as the other grains. As a result of the low water or completely water-free preparation, variations in quality due to different water contents of the pre-mixture are eliminated. In process step d), these characteristics of the process according to the invention provide the advantage that variations in the tablet hardness are reduced to a minimum. Whereas the usual pre-mixtures to be tableted provide formed bodies with a fixed compression pressure of the tableting press, which can vary in terms of their hardness by up to 30%, these variation ranges are substantially less than 10% in the case of the process according to the invention.

The process sequence of the process according to the invention is optimised since subsequent admixing of further constituents is eliminated. Finely particulate constituents which are to be used can "be bonded" to the large particles providing substantial advantages during dyeing of the particles, as detailed above. The incorporation of fragrances or other liquid constituents via "fragrance beads" or "active substance beads" leads to the migration tendency of these substances during compression being reduced to a minimum, this having a favourable effect on the properties of the formed bodies obtained.

Consequently, the detergent and cleaning agent formed bodies produced according to the process of the invention are stable in storage even in the unpackaged state, do not become hard subsequently and require no air tight individual packaging. Compared with conventional formed bodies, they are characterised by a greater hardness and better solubility so that they can be metered into domestic washing machines via the dispensing chamber. Moreover, they are stable when subjected to sudden stresses such as being dropped, tending neither to break nor to exhibit edge breakage phenomena.

Examples

Example 1:

In a batch mixer (20 litres) equipped with a knife head comminuter (chopper) further components, including a binder, were added to spray dried granules S1 and S2 (for

details of the composition compare table 1). The addition of the possibly present non-ionic surfactants liquid at temperatures below 45°C and a pressure of 1 bar was effected in the mixer by atomising through nozzles into the powder stream. The mixture was then homogenised for a further two minutes and subsequently passed to a double screw extruder whose granulation head had been preheated to temperatures between 50 and 65°C, preferably to 62°C. The pre-mixture was plasticised under the shearing effect of the extruder screw and subsequently extruded at a pressure of between 50 and 100 bar, preferably at around 78 bar through the perforated extruder head die to form fine strands of a diameter of 1.4 mm, which strands, after discharge from the die, were comminuted by means of a chopping knife to form approximately spherical granules (ratio of length to diameter approximately 1, hot chop effect). The warm granules thus obtained were rounded off for one minute in a commercial rounding device of the Marumerizer® type and, if necessary, coated with a finely particulate powder. The bulk density of the extrudates A1 and A2 thus produced was 800 ± 50 g/l, the particle sizes were entirely between 1200 and 1500 µm. The composition of this process end product from intermediate step a) is detailed in table 2.

Table 1: Composition of the spray dried granules (% by weight):

	S1	S2
C ₉ -C ₁₃ alkyl benzene sulphonate	26.3	12.07
Tallow fatty alcohol with an average of 5 EO	1.1	-
C ₁₂ -C ₁₈ sodium fatty acid soap	1.4	3.0
Sodium hydroxide	-	0.03
Sodium carbonate	9.4	4.15
Na-hydroxyethane-1,1-diphosphonate	-	0.8
Polyvinyl pyrrolidone	-	0.8
Copolymeric sodium salt of acrylic acid and maleic acid	4.0	4.15
Zeolite A, based on anhydrous active substance	39.5	57.75
Amorphous sodium disilicate	2.8	-
Water	13.6	16.65
Salts from solutions	Remainder	Remainder

Table 2: Composition of the process end products from intermediate step a) (% by weight)

	A1	A2
Spray dried granules	61.0 (S1)	65.71 (S2)
C ₁₂ -C ₁₈ fatty alkyl sulphate *	6.0	11.83
Copolymeric sodium salt of acrylic acid and maleic acid	3.0	2.96
Sodium perborate monohydrate	20.0	-
Polyethylene glycol (4000 g mol ⁻¹)	6.0	3.59
C ₁₂ -C ₁₈ fatty alcohol with an average of 7 EO	4.0	8.92
Trisodium citrate dihydrate	-	6.99

* Composition of the fatty alkyl sulphate:

5 in Example A1: 92.00% by weight of active substance, 3.70% by weight of sodium sulphate, 2.80% by weight of other salts from raw materials and unsulphonated fractions as well as 1.50% by weight of water.

10 in Example A2: 75% by weight C₁₂-C₁₈ alkyl sulphate, 17% by weight of sodium sulphate, 3% by weight of sodium carbonate, 1% by weight of water, remainder salts from solutions.

15 For the production of the process end products from intermediate step b), a soil release polymer, enzymes, defoamer and bleach activator were introduced into a Lödige ploughshare mixer and sprayed with perfume oil and an anhydrous swollen polymer while the mixing tool was operating. After an agglomeration time of 30 seconds, a powdering agent was added. After 30 seconds, the agglomerates were coated with the powdering agent. The addition of the cellulose-based disintegration aid took place last without an intense wetting with agglomeration liquid occurring. The agglomerates obtained had particle sizes of between 1000 and 1600 µm; their composition is detailed in Table 3.

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Table 3: Composition of the process end product from intermediate step b) (% by weight)

	B
Terephthalic acid ethylene glycol PEG ester	4.8
Enzyme mixture (protease, lipolase, cellulase, amylase)	11.3
Paraffin silicone defoamer, 15% on soda	18.2
Tetraacetylenediamine	33.4
Perfume oil	1.9
Hydroxypropyl starch, 5%, swollen in glycerine	1.9
Zeolite A (powdering agent)	1.9
Cellulose disintegrating agent	26.6

- 5 In process step c), 79.33% by weight of extrudates A1 was mixed with 20.67% by weight of granules B and subsequently compressed in process step d) on an eccentric press to form the formed bodies E1 according to the invention. The tablets had a diameter of 44 mm, a height of 17 mm and a weight of 36g.
- 10 The hardness of the tablets was measured by forming the tablets up to breaking point, the force being applied to the lateral surfaces of the tablet, determining the maximum force which the tablet withstood.
- 15 To determine the tablet disintegration, the tablets were placed into a glass beaker containing water (600 ml water, temperature: 30°C) and the time which passed up to complete tablet disintegration was measured.
- 20 To determine the residual behaviour and/or the solubility behaviour (solubility test), one tablet was introduced into one litre water in a 2 litre glass beaker with stirring (800 rpm, with laboratory stirrer/propeller stirrer head, centred at a distance of 1.5 cm from the glass beaker bottom) and stirred for 1.5 minutes at 30°C. The test was carried out with water of 16° German hardness. Subsequently, the wash liquor was

poured through a sieve (80 μm). The glass beaker was rinsed with very little cold water over the sieve. A double determination was carried out. The sieves were dried in the drying cabinet at $40^{\circ}\text{C} \pm 2^{\circ}\text{C}$ until a constant weight was reached and the detergent residue was weighed. The residue is indicated as the mean value of the two individual determinations, in %. In the case of deviations between the individual results by more than 20%, further experiments are usually carried out; however, this was not necessary in the case of the present investigations.

The physical properties of the detergent tablets are detailed in Table 4.

Table 4: Detergent tables (physical data)

	E1
Fracture hardness (N)	50
Disintegration time(s)	12
Dissolution test (residue) (% by weight)	18
Drop test from a height of 1 m	Stable, no edge disintegration

Example 2:

By mixing the individual components in a Lödige ploughshare mixer (process step c)), two pre-mixtures were produced which were subsequently compressed into two layer tablets E2 by sequential filling of the matrices of an eccentric press. The proportion of blue phase of the tablet was 25%, the proportion of white phase 75%. The extrudates A1 described above were used as end product from partial step a), the process end product from partial step b) is obtained from the details in table 5 in that all constituents, with the exception of extrudates A1, were agglomerated in a mixer granulator. The particle size of these agglomerates was between 1200 and 1400 μm .

The composition of the individual phases is indicated in Table 5.

Table 5: Composition of the double layer tablets (broken down according to layers)
(% by weight)

	Blue phase	White phase
Terephthalic acid ethylene glycol PEG ester	-	1.32
Enzyme mixture (protease, lipolase, cellulase, amylase)	9.35	-
Paraffin silicone defoamer, 15% on soda	3.75	3.75
Tetreaacetylenediamine	-	9.21
Fragrance beads *	4.00	4.00
Hydroxypropyl starch, 5%, swollen in glycerine	0.40	0.40
Extrudate A1	76.60	75.24
Zeolite A (powdering agent)	-	0.40
Zeolite A (powdering agent), dyed blue	0.40	-
Cellulose disintegrating agent	-	5.50
Cellulose disintegrating agent, dyed blue	5.50	-

- 5 * Fragrance beads produced according to the theorem of the earlier German patent application 197 46 780.6, containing 10% perfume.

10 Process step d) was carried out by introducing first the pre-mixture for the white phase and subjecting it to light preliminary compression followed by the pre-mixture for the blue phase and fully compressing the formed body.

The physical properties of the double layer detergent tablets are shown in Table 6.

Table 6: Detergent tablets (physical data)

	E2
Fracture hardness (N)	60
Disintegration time(s)	6
Dissolution test (residue) (% by weight)	18
Drop test from a height of 1 m	Stable, no edge disintegration

5 **Example 3:**

In an analogous manner as for example 2, double layer detergent and cleaning agent formed bodies were produced, the extrudates A2 described above being used as the end product from process step a). The composition of the process end product from step b) was also different in this case and is in turn shown in the following table, in
 10 which all components, with the exception of A2, were granulated to form particles with particle sizes of between 1200 and 1400 μm .

In contrast to example 2, the bleaching agent is introduced into the process in this example not via the product of step a) but via step b). The composition of the
 15 individual phases is shown in Table 7.

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Table 7: Composition of the double layer tables (broken down according to layers)
(% by weight)

	Blue phase	White phase
Terephthalic acid ethylene glycol PEG ester	1.00	1.00
Enzyme mixture (protease, lipolase, cellulase, amylase)	2.35	2.35
Paraffin silicone defoamer, 15% on soda	3.75	3.75
Tetraacetylenediamine, dyed blue	21.00	-
Sodium percarbonate	-	21.00
Fragrance beads *	4.00	4.00
Hydroxypropyl starch, 5%, swollen in glycerine	0.60	0.60
Extrudate A2	61.50	61.50
Zeolite A (powdering agent)	-	0.30
Zeolite A (powdering agent), dyed blue	0.30	-
Cellulose disintegrating agent	-	5.50
Cellulose disintegrating agent, dyed blue	5.50	-

- 5 * Fragrance beads produced according to the theorem of the earlier German patent application 197 46 780.6, containing 10% perfume.

10 Process step d) was carried out in an analogous manner to example 2 by introducing first the pre-mixture for the white phase and subjecting it to light preliminary compression followed by the pre-mixture for the blue phase and fully compressing the formed body E3.

The physical properties of the double layer detergent tablets are shown in Table 8.

15

Table 8: Detergent tablets (physical data)

	E3
Fracture hardness (N)	50
Disintegration time(s)	10
Dissolution test (residue) (% by weight)	16
Drop test from a height of 1 m	Stable, no edge disintegration

Claims:

1. A process for the production of detergent and cleaning agent formed bodies comprising the steps of
 - 5 a) preparing mainly solid, finely particulate constituents in a plasticised state to form pourable and flowable high density granules,
 - b) preparing one or several agglomerates from further constituents to be optionally added,
 - 10 c) combining the granules and agglomerates of steps a) and b) to form compressed pre-mixtures, and
 - d) compressing the pre-mixtures to form single or multiple phase formed bodies, wherein the granules and agglomerates produced in steps a) and b) are essentially free from fines and have particles sizes of between 800 and 2000 μm .
- 15 2. A process as claimed in claim 1, wherein the granules produced in steps a) and b) consist in a proportion of at least 75% by weight of particles with particle sizes of between 800 and 1600 μm .
- 20 3. A process as claimed in claim 1 or 2, wherein the granules and agglomerates produced in steps a) and b) consist in a proportion of at least 60% by weight of particles with particle sizes of between 1200 and 1500 μm .
- 25 4. A process as claimed in any of claims 1 to 3, wherein the pourable and flowable high density granules in step a) are prepared by mixing detergent or cleaning agent compounds and/or detergent or cleaning agent raw materials, and simultaneously or subsequently forming, a solid pre-mixture which contains individual raw materials and/or compounds which are solid at room temperature and a pressure of 1 bar and exhibit a melting point or softening point of not less than 45°C, the pre-mixture also containing as required, up to 30 10% by weight of non-ionic surfactants which are liquid at temperatures below 45°C and a pressure of 1 bar and which, by applying compaction forces

at temperatures of at least 45°C are converted into a grain and, when required are subsequently processed or treated further, with the proviso that

- the pre-mixture is essentially anhydrous and
- at least one raw material or compound, which is solid at a pressure of 1 bar and temperatures below 45°, is present under the processing conditions as a melt, this melt serving as a polyfunctional, water-soluble binder which, during the production of the agents, operates as a slip and as an adhesive for the solid detergent or cleaning agent compounds or raw materials but has a disintegrating effect during the re-dissolution of the agent in aqueous liquor.

10

5. A process as claimed in any of claims 1 to 3, wherein the production of the pourable and flowable high density granules of step a) takes place by initially producing a solid pre-mixture which contains at least one non-aqueous binder and a solid raw material or solid raw materials from a raw material class, which material is present as a solid at room temperature and a pressure of 1 bar and has a melting and/or softening point of not less than 45°C, in quantities of at least 50% by weight, and the pre-mixture is converted into a grain by applying compaction forces at temperatures of at least 45°C and subsequently, when required subjecting the grains to further processing or treatment with the proviso that

20

- the pre-mixture is essentially anhydrous and
- at least one non-aqueous binder which is solid at a pressure of 1 bar and temperatures below 45° but is present under the processing conditions as a melt, this melt serving as a polyfunctional, water-soluble binder which, during the production of the agents, operates as a slip and as an adhesive for the solid raw materials but has a disintegrating effect during the re-dissolution of the agent in the aqueous liquor.
- and a bulk density of at least 600 g/l is adjusted.

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30

6. A process as claimed in any of claims 1 to 3, wherein the production of the pourable and flowable high density granules in step a) is effected by mixing detergent or cleaning agent compounds and/or detergent or cleaning agent raw

materials with simultaneous or subsequent forming, a solid pre-mixture being initially produced which contains individual raw materials and/or compounds which are present as a solid at room temperature and a pressure of 1 bar, this pre-mixture being subsequently converted into a grain by the application of compaction forces and, when required, subsequently being further treated or processed, with the proviso that the pre-mixture is essentially anhydrous and a forming aid is used which is liquid under the forming conditions, in particular also at room temperature and a pressure of 1 bar and is used in the form of a polymer swollen in a non-aqueous or low water solution.

5

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7. A process as claimed in any of claims 1 to 6, wherein the production of agglomerates in step b) takes place essentially free from water.

15

8. A process as claimed in any of claims 1 to 7, wherein the production of the agglomerates takes place with an addition of plasticiser and/or slip agent.

9. A process as claimed in claim 8, wherein polyethylene glycols or ethoxylated alcohols are used as plasticiser and/or slip agent.

20

10. A process as claimed in claim 8, wherein anhydrous swollen polymers are used as plasticiser and/or slip agent.

25

11. A process as claimed in any of claims 1 to 10, wherein the agglomerates produced in step b) are obtained by bonding the more finely particulate granules to those obtained in step a).

30

12. A process as claimed in any of claims 1 to 11, wherein the proportion of granules produced in step a) present in pre-mixture c) is 40 to 95% by weight, based on the pre-mixture.

13. A process as claimed in claim 12, wherein the proportion is 50 to 90% by weight.

14. A process as claimed in claim 12, wherein the proportion is 60 to 85% by weight.
- 5 15. A process as claimed in any of claims 1 to 14, wherein at least one of the agglomerates produced in step b) contains enzyme(s).
- 10 16. A process as claimed in claim 15, wherein at least one of the agglomerates produced in step b) contains granulated enzyme(s) with particle sizes above 700 μm .
17. A process as claimed in any of claims 1 to 16, wherein at least one of the agglomerates produced in step b) contains perfume.
- 15 18. A process as claimed in any of claims 1 to 17, wherein at least one of the agglomerates produced in step b) contains a cellulose-based disintegration aid.
- 20 19. A process as claimed in claim 18, wherein at least one of the agglomerates produced in step b) contains a compacted or anhydrous agglomerated cellulose-based disintegration aid which consists in a proportion of at least 90% by weight of particles with sizes above 400 μm .
- 25 20. A process as claimed in claim 19, wherein the proportion is at least 66% by weight of particles with sizes above 800 μm .
21. A process as claimed in claim 19, wherein the proportion is at least 50% by weight of particles with sizes above 1200 μm .
- 30 22. A process as claimed in any of claims 1 to 21, wherein, in process step d) compression pressures are adjusted which lead to formed bodies with fracture hardness values of 20 to 150 N.

23. A process as claimed in claim 22, wherein the values are 40 to 100 N.
24. A process as claimed in claim 22, wherein the values are 50 to 80 N.
- 5 25. A process as claimed in any of claims 22 to 24, wherein multiple phase detergent and cleaning agent formed bodies are produced, the fracture hardness of the individual phases differing by maximum $\pm 15\%$.
26. A process as claimed in claim 25, wherein the maximum is $\pm 10\%$.
- 10 27. A process as claimed in any of claims 1 to 26, wherein the detergent and cleaning agent formed bodies have a weight of between 10 and 150 g.
28. A process as claimed in claim 27, wherein the weight is between 20 and 100 g.
- 15 29. A process as claimed in claim 27, wherein the weight is between 35 and 75 g.